INEEL/EXT-98-01108

November 1998

Mixed Waste Focus Area/ Characterization Monitoring Sensor Technology Nondestructive Waste Assay Capability Evaluation Project

End-User Summary Report

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Prepared for the
U.S. Department of Energy

Under DOE Idaho Operations Office
Contract DE-AC07-94ID13223

SUMMARY

The Mixed Waste Focus Area (MWFA) in conjunction with the Characterization Monitoring and Sensor Technology (CMST) crosscut program identified the need to objectively evaluate the capability of nondestructive waste assay (NDA) technologies. This was done because of a general lack of NDA technology performance data with respect to a representative cross section of waste form configurations comprising the Department of Energy (DOE) contact-handled alpha contaminated [e.g., transuranic (TRU) waste]. The overall objective of the Capability Evaluation Project (CEP) was to establish a known and unbiased NDA data and information base that can be used to support end-user decisions with regards to technology system selection and to support technology development organizations in identifying technology system deficiencies.

The CEP was conducted at the Idaho National Engineering and Environmental Laboratory (INEEL) Radioactive Waste Management Complex (RWMC) from September 1997 through May 1998, which required that all participating technologies be on a mobile platform. The CEP evaluated assay system performance parameters on TRU material quantification contained in 55-gallon type waste containers for five waste NDA technologies. Program participants included Bio-Imaging Research, Inc., (BIR), Canberra Industries, Inc., and the Los Alamos National Laboratory (LANL). The NDA technologies provided by these participants represent those commonly employed at waste management characterization facilities with the exception of the active neutron assay technique. Despite the lack of participation of an active neutron based waste NDA technology, the CEP objective of establishing a capability baseline was not significantly compromised.

The evaluation was performed by presenting project participants a set of well-characterized test samples in a blind test format. The test samples consisted of surrogate and actual TRU waste containers. Test sample measurement data were acquired from each participant through a protocol that accounted for test sample quality assurance and confidentiality. Results were evaluated per criteria and performance assessment mechanisms founded in the Department of Energy Carlsbad Area Office (DOE-CAO), Quality Assurance Program Plan (QAPP) for the Waste Isolation Pilot Plant (WIPP).

The primary performance parameters evaluated in the CEP were measurement bias and relative precision. The performance of a given NDA technology is a direct function of the attributes represented by the waste matrix configuration. Such attributes include matrix density, matrix elemental composition, radionuclidic composition, radionuclide mass loading, and the spatial variation of these components. Analyzing the manner in which bias and precision vary as a function of test sample attribute and NDA technology provides a foundation for deriving performance capability and limitation statements and determines which waste matrix attributes, or combinations of attributes, are compatible or incompatible with existing technologies.

The data indicate that the NDA systems evaluated have a definite capability to perform assay of contact-handled TRU waste packaged in 55-gallon drums, which exhibit reasonable matrix densities and radionuclide loadings, within the acceptance

bounds of the QAPP and Performance Demonstration Program (PDP) criteria. Technologies employed to account for matrix effects on quantitative assay processes appear sufficiently developed for waste matrices exhibiting reasonable densities, e.g., less than 0.6 g/cm^3. Additionally, radioactive material mass loadings comprised of nominal weapons grade plutonium sufficient to yield statistically significant data, within a reasonable measurement period, are within the current technology capability envelope. This indicates that in general, calibration, data acquisition and reduction techniques under such conditions, are adequate with respect to the bias and precision performance quality assurance objectives (QAOs).

Performance with respect to bias and precision parameters and minimum detectable concentration (MDC) criteria for test samples in the vicinity of the 100 nCi/g low-level waste (LLW)/TRU segregation point is questionable. To a certain extent this can be mitigated through the use of longer measurement times. In other instances, there does not appear to be adequate accounting of interferences affecting the MDC, such that increased measurement times leads to diminishing returns.

Finally, the ability to yield acceptable bias and precision performance is, under many circumstances, compromised by the number of complicating attributes inherent in the waste matrix configuration. For instance, when the radionuclidic distribution departs from that associated with nominal weapons grade plutonium, there is reasonable capability to correctly determine the mass of the various nuclides. If this configuration is compounded by a low mass of the primary plutonium isotope of quantitation, the ability to achieve acceptable performance deteriorates. If this configuration is again compounded with a high density matrix, the ability to perform in an acceptable manner is further reduced.

In summary, the CEP achieved the stated end-user objective. The data indicate that the nondestructive waste assay systems evaluated have a definite capability to perform assay of contact-handled TRU waste packaged in 55-gallon drums. There is, however, a performance envelope where this capability exists, an area near the envelope boundaries where it is questionable, and a realm outside the envelope where the technologies do not perform. Therefore, the end user must be aware of this envelope and ensure the appropriate technology is selected. This program provides the end user with waste type specific performance data to assist in the assessment and selection of a given waste NDA technology. Additionally, the CEP afforded the private sector participants the opportunity to evaluate system performance using National Institute of Standards and Technology (NIST) traceable radioactive standards and actual TRU waste. This enabled several participants to make significant enhancements to their respective systems and supported all participants in attaining DOE-CAO certification. Ultimately, the DOE end users will benefit from these enhancements.

ACKNOWLEDGMENTS

The magnitude of the Capability Evaluation Project necessitated the participation and support of numerous individuals. Special acknowledgment is due to the invaluable technical contributions of Kenneth Coop, Gary Twedell, and Paul Hurley. Additionally, physical implementation and operational coordination could not have been accomplished without the dedicated efforts of James Pletscher, Douglas Oakey, and Robert Mahoney. This work was supported by the U. S. Department of Energy, Office of Science and Technology through the Mixed Waste Focus Area, and Characterization, Monitoring, and Sensor Technology Crosscut, under Idaho Operations Office Contract DE-AC07-94ID13223.

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TABLES

ACRONYMS

BIR	Bio-Imaging Research
CB	confidence bounds
CEP	Capability Evaluation Project
CMST	Characterization Monitoring and Sensor Technology
DOE	Department of Energy
DOE-CAO	Department of Energy-Carlsbad Area Office
DOT	Department of Transportation
HENC	High Efficiency Neutron Counter
IDC	item description code
INEEL	Idaho National Engineering and Environmental Laboratory
LANL	Los Alamos National Laboratory
LLW	low-level waste
MDC	minimum detectable concentration
MWFA	Mixed Waste Focus Area
NAS	Neutron Assay Software
NDA	nondestructive waste assay
NIST	National Institute of Science and Technology
PDP	Performance Demonstration Program
QAO	quality assurance objective
QAPP	Quality Assurance Program Plan
RF	Rocky Flats
RFETS	Rocky Flats Environmental Technology Site
RWMC	Radioactive Waste Management Complex
SAS	SWEPP nondestructive assay system
SG	Surrogate
SGS	Segmented Gamma Scanner
SWEPP	Stored Waste Examination Pilot Plant
TGS	Tomographic Gamma Scanner
TRU	transuranic
WIPP	Waste Isolation Pilot Plant
WIT	Waste Inspection Tomography
WRM	Working Reference Material

Mixed Waste Focus Area/Characterization Monitoring Sensor Technology Nondestructive Waste Assay Capability Evaluation Project End-User Summary Report

1. INTRODUCTION

The Mixed Waste Focus Area (MWFA) in conjunction with the Characterization Monitoring and Sensor Technology (CMST) crosscut area program identified the need to establish a mechanism to objectively evaluate the utility of waste assay system technologies. The need for the evaluation was based on the general lack of nondestructive waste assay system performance data with respect to a representative cross section of waste form configurations comprising the Department of Energy (DOE) transuranic (TRU) contaminated waste inventory. This situation compromised the ability of the potential end user to adequately assess the viability of available technologies in terms of waste form characterization and compliance at their respective sites. It also complicated the effort to clearly identify deficiencies in the existing technology base that need to be revealed to facilitate development resource allocation.

The Capability Evaluation Project (CEP) was conducted at the Idaho National Engineering and Environmental Laboratory (INEEL) Radioactive Waste Management Complex (RWMC) from September 1997 through May 1998, which required that all participating technologies be on a mobile platform. The CEP evaluated assay system performance parameters on TRU material quantification contained in 55-gallon type waste containers for five waste NDA technologies. Program participants included Bio-Imaging Research, Inc., (BIR), Canberra Industries, Inc., and the Los Alamos National Laboratory (LANL). The NDA technologies provided by these participants represent those commonly employed at waste management characterization facilities with the exception of the active neutron assay technique. Despite the lack of participation of an active neutron based waste NDA technology, the CEP objective of establishing a capability baseline was not significantly compromised.

The evaluation was performed by presenting the project participants a set of well-characterized test samples. Test sample measurement data were acquired from each participant through a protocol that accounted for test sample quality assurance and confidentiality. Results were evaluated per criteria and performance assessment mechanisms founded in the Department of Energy Carlsbad Area Office (DOE-CAO) Transuranic Waste Characterization Quality Assurance Program Plan (QAPP).[1] The CEP test addressed performance for TRU waste contained in the standard 55-gallon container only.

1.1 Objective

The objective of the CEP was to establish a known and unbiased nondestructive waste assay data and information base that can be used to support the end user in terms of available capability and identify technology deficiencies for development consideration. Capability and deficiency are defined in terms of the ability to comply with applicable requirements and quality assurance objectives (QAOs) of the DOE-CAO QAPP. To ensure CEP generated data are useful per this stipulation, the project was structured to provide data supportive of compliance determination processes. This was accomplished by adapting DOE-CAO QAPP requirements for system performance evaluation. By maintaining an identifiable relationship between CEP and DOE-CAO criteria/evaluation protocols, nondestructive waste assay capability and deficiency statements, as a function of waste type and/or characteristic, can be derived per an established baseline. Consistent with this objective, the CEP content, conduct, data acquisition, and evaluation techniques are delineated in the project test plan, Nondestructive Assay System Capability Evaluation Project Test Plan for Transuranic Contaminated Waste Forms.[2] Hence, the CEP provides a platform to establish system performance for alpha contaminated waste forms related to DOE-CAO requirements in an objective documented manner.

2. CEP PERFORMANCE EVALUATION CRITERIA AND TEST CONDUCT

The CEP performance evaluation criteria and test conduct are described in this section. Criteria used to benchmark performance were adapted from existing DOE-CAO requirements and used to define acceptable or unacceptable performance for two primary parameters: total bias and relative precision on total TRU alpha activity. Additionally, the data acquisition procedure is critical to the quality of acquired data, confidentiality of test sample contents, and integrity of the final evaluated performance parameters. Hence, a brief discussion of the physical control of test samples, quality assurance measures, and participant reporting requirements is included.

2.1 CEP Performance Evaluation Criteria

Criteria used to evaluate assay system capability were adapted from the DOE-CAO Performance Demonstration Program Plan for Nondestructive Assay for the TRU Waste Characterization Program[3] founded in and promulgated by the QAPP. The QAOs for total bias and relative precision for the noninterfering and interfering matrix are tabulated in Tables 1 and 2. The Nondestructive Assay Performance Demonstration Program (NDA PDP) noninterfering and interfering matrix total bias QAOs are shown in Table 1. The noninterfering alpha activity, range-specific QAOs were used for test samples exhibiting a matrix configuration considered not to represent a significant complication, e.g., high density, to an NDA system. Likewise, the interfering, total bias, QAOs were employed for test samples with matrix configurations that manifest a complication to the NDA system, e.g., high density. The low and high bias QAOs are the limits on the two-sided 95% confidence bounds (CB) for the ratio of the mean measured to the known or accepted reference value expressed as a percent.

The NDA PDP noninterfering and interfering upper maximum measured relative precision QAOs are tabulated in Table 2, Columns 3 and 4, respectively. The maximum measured precision values are based on the upper one-sided 95% confidence of a precision determination exactly equal to the QAPP based QAO values in Column 2. The precision QAO limits have been modified from the NDA PDP values to account for the enhanced number of replicates, i.e., eight compared to six, used in the CEP.

Table 1. NDA PDP Bias QAOs.

Activity Range (α-Curies [a])	Instrument Bias QAO Values for $\%R_L$ and $\%R_U$ (noninterfering)[b]	Total Bias QAO Values for $\%R_L$ and $\%R_U$ (interfering)
>0 to 0.02	Low: 75% High: 125%	Low: 40% High: 175%
>0.02 to 0.2	Low: 50% High: 150%	Low: 30% High: 200%
>0.2 to 2.0	Low: 50% High: 150%	Low: 30% High: 200%
>2.0	Low: 75% High: 125%	Low: 50% High: 150%

a. Applicable range of TRU activity in a 208-L (55-gallon) drum to which the QAOs apply; units are curies of alpha emitting TRU isotopes with half-lives greater than 20 years.

b. Limits on the two-sided 95% confidence bound for the ratio of the mean of the measured values to the known (or accepted) value, expressed as a percent.

Table 2. Measured relative precision QAOs adjusted for eight replicates.

Activity Range in Curies[a]	Maximum Allowable Precision[b] (95% CB of QAPP QAO)	Maximum Measured Precision (%RSD)[c] @ 8 replicates (noninterfering)	Maximum Measured Precision (%RSD)[c] @ 8 replicates (interfering)
>0 to 0.02	29.2	$\leq$ 16.0	$\leq$ 18.0
>0.02 to 0.2	21.9	$\leq$ 12.0	$\leq$ 14.0
>0.2 to 2.0	14.6	$\leq$ 8.0	$\leq$ 14.0
>2.0	7.3	$\leq$ 4.1	$\leq$ 7.0

a. Applicable range of TRU activity in a 208-L (55-gallon) drum to which the QAOs apply; units are curies of alpha emitting TRU isotopes with half-lives greater than 20 years.

b. QAPP QAO limit for the measured precision corresponding to a 95% upper confidence bound on true system precision.

c. Measured precision that must be met to satisfy the same precision criteria as the QAPP QAOs, except based on eight replicates. The values are one relative standard deviation referenced to the known (or accepted) value for the test, s/μ.

2.2 CEP Test Conduct

The CEP was conducted using a blind test format. Participants in the program were presented with 32 test samples for which the matrix configuration and radioactive material loading were maintained confidential throughout the duration of the CEP. Participants were provided the matrix type [item description code (IDC) of the test sample] and the net weight only. The participants then processed the test sample through their routine NDA procedure per the CEP declared hardware configuration and software version. Participants were not allowed to change this predeclared configuration throughout the duration of testing. Each participant was given 6 weeks to process as many of the 32 test samples as possible.

2.2.1 CEP Personnel

There were four significant test implementation personnel designated to implement the various functions of the CEP: project referee, RWMC representative, RWMC sample attestant, and participant assay system representative. The functions and responsibilities of these individuals are very similar, if not identical, to designees in the NDA PDP program.

The project referee was responsible for the specification of the test sample set and the evaluation technique. A description of the test sample set is included in Section 3. The RWMC representative was responsible for the staging of test samples, preparing required QA documentation, and ensuring confidentiality of the test samples. The RWMC sample attestant performed the function of an independent quality assurance check and verified and signed off on the loading and unloading of radioactive standards into the surrogate test samples, as well as chain-of-custody documentation. The RWMC representative ensured that test samples were delivered to the participant assay system representative and maintained a chain-of-custody log for each test sample until the sample was returned. The RWMC representative was responsible for acquiring test measurement data/information from the participant and transmitting it to the project referee. All CEP personnel maintained confidentiality of test sample configurations and associated documentation throughout the duration of the CEP project. Details on the responsibilities of each of the project positions, as well as required documentation of the procedure, are delineated in the CEP test plan.

2.2.2 Analytical and Data Reporting Requirements

After receipt of a test sample, replicate measurements were performed per the participant declared system configuration. Data for the evaluation of precision and bias were acquired through eight replicate measurements of each test sample. The test sample was required to be indexed or otherwise repositioned between replicate measurements. On completion of the eight replicate test sample measurement series, the participant assay system representative transferred, at a minimum, a hard-copy report containing the measurement results to the RWMC representative. All assay measurement data were logged and transmitted to the project referee. Reports consisted, at a minimum, of the following information for each test sample replicate:

- Measurement system to which the report pertains and an associated software/hardware configuration identifier
- Test sample for which the data are being reported
- Method used to determine the quantity of each isotope, e.g., gamma mass ratio measurement, application of constants, etc.
- Replicate number corresponding to the analytical data for each eight test sample replicate set
- Identity and activity (alpha Ci) plus 2σ error estimate for each radioisotope and radionuclide identified in each replicate test sample, either measured directly or determined from other means such as mass ratios
- Total ^{239}Pu fissile gram equivalent (g) and associated 2σ error estimate for each replicate test sample
- Total alpha activity and associated 2σ error estimate for each replicate test sample
- Thermal power and associated 2σ error estimate
- Total measurement duration time per assay.

2.2.3 Test Apparatus Quality Control

Test samples used in the execution of the CEP project was required to have pedigrees and/or documentation sufficient to establish a level of quality commensurate with the objectives of the project. CEP test samples consisted of surrogate matrix drums, radioactive material standards, hereafter referred to as Working Reference Materials (WRMs), and actual waste Rocky Flats Environmental Technology Site (RFETS) drums. The technique for establishing actual RFETS waste test sample radioactive material mass and uncertainty estimate will be addressed in the Mixed Waste Focus Area/Characterization Monitoring Sensor Technology Nondestructive Waste Assay Capabilities Evaluation Program - Final Report.[4] Quality control requirements for the surrogate matrix drums and WRMs are discussed in this section.

For the purpose of this project, the WRMs used to configure surrogate test samples, where practicable, are traceable to a certificate or other documentation issued by a technically competent certifying body such as the New Brunswick Laboratory or the National Institute of Standards and Technology (NIST). Nearly all WRMs used in the project met this qualification. Those standards that did not possess pedigrees of traceability had sufficient characterization data to establish confidence in the use of the WRM in the project. Certificates of traceability and/or adequate characterization data for each standard used in the project were placed in the project file for reference, as appropriate.

Surrogate drums are not per se traceable entities. Detailed documentation on the specification, design, and fabrication was adequate to establish the validity of such devices for use in the project. Documentation of the design and as-built configuration for each surrogate matrix drum employed in the project were placed in the CEP project files.

2.2.4 Test Limitations

As with all performance evaluation programs, it is important to note any aspects that may limit the application and use of data derived from the test process. The first point to note is that the test sample set is not all inclusive with respect to the DOE TRU waste inventory. Despite the best efforts to specify and select test samples, it is not possible to bound all configurations resident in the inventory. Therefore, extrapolation of performance beyond the established attributes of the test sample set should be performed with the assistance of a waste NDA expert. Second, the use of surrogate test samples, although precisely known in terms of their properties and radioactive material mass loadings, are only applicable to the extent they approximate nominal characteristics of inventory subpopulations. Additionally, though a large number of combinations can be realized with the available set of matrix surrogate and radioactive standards, certain parameters of interest cannot be tested. The WRM set at the RWMC Stored Waste Examination Pilot Plant (SWEPP) facility allowed a reproduction of most nominal radionuclidic/isotopic compositions found in the DOE complex inventory, but not the entire spectrum.

Although the surrogate test samples were precisely known in all aspects, they have the drawback of possessing one effective matrix material configuration. An advantage of using actual waste drums as test samples is that additional realistic configurations can be added to the test sample set. Actual waste test samples do have the drawback in that the radioactive material loading and physical distribution has associated uncertainty. This requires the determination of time consuming, best estimates of mass and the complication of accounting for this uncertainty in the evaluation process. Nevertheless, the actual waste test samples contribute an added dimension and robustness to the CEP test process.

Another limitation regarding the testing of system parameters of interest is the time duration of the testing sequence itself. The number and type of actual test samples that were processed through a given waste NDA system was limited by the allowable test plan duration. The test samples that are included represent the envelope of the capability evaluation and therefore roughly define the space for which statements of utility and capability can be made.

It was recognized that the scope of the CEP would not provide all the data necessary to completely assess the ability to demonstrate compliance with applicable NDA requirements and criteria. However, the CEP did yield capability information regarding key functional parameters related to waste type, which the system/technology must be able to accommodate.

3. CEP TEST SAMPLE INFORMATION

CEP participant performance data were acquired through the use of surrogate and actual waste type test samples representative of a cross section of the DOE TRU contaminated waste inventory. Test sample specification and selection were determined based on waste types comprising a predominant fraction of the inventory and those for which there is particular interest in establishing waste NDA capability data. Determination of the test sample set also considered the various attributes associated with each waste category. The occurrence frequency and magnitude of these attributes are for the most part a function of the waste category. Attributes types include: TRU alpha activity loading/concentration, radioactive material distribution/physical form, radionuclidic/isotopic composition, chemical composition/matrix compound of radioactive material, general radiation emission characteristics of waste type, elemental composition of waste matrix, density of matrix (average and spatial dependent), and packaging configuration of waste matrix. The intent of test sample specification was to ensure representation of as many attributes and variations as possible, therefore, multiple test samples were indicated for each waste category. The resulting test sample set included at least one surrogate and up to three actual waste type test samples per waste type category. This resulted in a test sample set containing 32 samples.

The test samples specified for use in the CEP were of two distinct types: surrogate and actual waste. Each of the test sample types had unique features that contribute to the scope of the CEP evaluation process. Surrogate test samples were precisely known in terms of both the matrix and radioactive material properties. This allows for precise determination of waste NDA capability in terms of the bias and precision parameters and an exact knowledge of the sample contents for detailed analysis of systems response measures. Actual waste test samples are completely realistic over all attributes and to the extent they are representative of their respective subpopulations serve as excellent test samples. The drawback associated with the use of actual waste test samples is that certain attributes, in particular, accurate knowledge of the mass of each entrained radionuclide, must be estimated for evaluation purposes. Nevertheless, their inclusion in the test sample set is important in that surrogate test samples are limited relative to the approximation of actual waste form configurations.

3.1 Surrogate Test Sample Description

Surrogate test samples were derived from apparatus that could be configured to represent a given subset of waste form variables and combinations thereof. Such test samples were realized through an appropriate combination of NIST traceable WRMs with simulated waste matrices of known composition/configuration installed in 55-gallon Department of Transportation (DOT) 17C type drum containers. This allowed for the production of representative waste form configurations with precisely known radioactive material loadings and matrix configurations. These configurations allowed specific measurement bias sources to be evaluated.

Eleven different surrogate matrix drums were employed in the CEP. The surrogate matrix drums by waste type are graphite, combustibles, filter/insulation, inorganic sludge, organic sludge, molten salts (two configurations), glass, raschig rings, metals, and an empty or zero matrix drum. Surrogate drums simulate nominal actual waste characteristics such as elemental composition, density, and packaging configuration. The matrix materials are fixed to an internal support structure to ensure the matrix configuration does not change with time. The internal support structure also accommodates the insertion and precise location of WRMs to produce the desired test sample. Through the use of the surrogate insert tubes and WRM insert fixtures, precise positioning and replication of a given surrogate configuration was achieved for each of the participants.

3.2 Actual Waste Test Samples

To complement the surrogate type test sample set, 21 actual waste containers were identified from the RWMC accessibly stored inventory for use in the CEP. These actual waste test samples were selected from an inventory of drums generated as a by-product of operations at the DOE Rocky Flats Plant. The waste types included in the selected set were inorganic sludge (IDC 001), organic sludge (IDC 003), graphite (IDC 300), dry combustibles (IDC 330), moist combustibles (IDC 336), cemented insulation/filter media (IDC 376), molten salts (IDC 409), electrorefining salt (IDC 411), glass (IDC 440), Raschig rings (IDC 442), nonspecial source metals (IDC 480), and leached nonspecial source metals (IDC 481). The selected actual RFETS waste containers are a reasonable representation of the waste type subpopulation based on available data regarding configurations and attributes of interest to the CEP. Although it is acknowledged that there are outliners in terms of anomalous waste form configurations within each waste type category, time constraints on the CEP do not allow a capability assessment of all possible configurations in the inventory.

To support assay system capability statements relative to actual waste forms, it was necessary to have a bounded estimate of the contents, including both the radioactive and matrix composition and configuration. There exists sufficient information with respect to the matrix and entrained radioactive material to support their use as CEP performance evaluation test samples. The specification of an actual RFETS test sample radioactive material mass and uncertainty was a detailed process and unlike the surrogate test sample has an associated uncertainty. A variety of data were used to establish a best estimate for the radioactive material mass loadings. Most important to this process was the use of data acquired via optimized data acquisition parameters determined as a function of waste configuration and radioactive material emission properties using the RWMC SWEPP nondestructive assay system (SAS). Additionally, INEEL NDA system uncertainty analysis information and data were of significance in arriving at a best estimate. The actual waste sludge type test samples also had radiochemistry data acquired from core samples taken from these specific test samples.

The test sample configurations and attributes form the basis and significance of the CEP test. As waste NDA system performance over the spectrum of possible waste form configurations was the primary interest of the CEP, the specific attributes and their configuration in each sample are of fundamental importance. It is the ability of a given waste NDA technology to accommodate the indicated attributes and configurations that is of interest to the end user and agencies responsible for resolving technology deficiencies. To assist interested parties in evaluating system performance and utility, certain attributes for each test sample were tabulated. The radioactive material constituent mass loading, TRU alpha activity, and the associated alpha activity concentration in units of TRU alpha activity nCi/g for each sample are of obvious importance. Tabulation of such data is presented in separate tables for the surrogate and actual waste type samples. Separation into surrogate and actual sample types for the radioactive material loading attribute is performed solely for the purpose of maintaining NIST traceable loadings from best estimate loadings that have uncertainty. Tables 3 and 4 contain radioactive material mass loadings, alpha activity, and alpha activity concentration values for the surrogate and actual waste test samples, respectively. Note that for three of the five actual waste sludge test samples there are two radioactive material mass loadings, alpha activity, and alpha activity concentration values. The second set of values were derived from statistically representative intrusive sampling procedures and radiochemistry based analysis of the samples. The radiochemistry data were used in the evaluation process and served as an important validation of the SAS generated mass estimates for the three sludge test samples.

Table 3. Surrogate test sample radioactive material loadings.

Surrogate Test Samples	Total TRU Activity (Ci) {α nCi/g}	^{239}Pu Mass (g)	^{241}Am Mass (g)	^{235}U Mass (g)	^{238}U Mass (g)	^{234}U Mass (g)
Graphite (SG1)	0.007 {73}	0.086	9.340e-05	—	—	—
Combustibles (SG2)	0.314 {7,149}	3.794	0.004	—	—	—
Filters/insul (SG3)	4.891 {1.22e5}	49.402	0.288[a]	—	—	—
Inorganic sludge (SG4)	0.084 {694}	0.154	0.020	8.201	3,827.6	0.038
Organic sludge (SG5)	0.077 {508}	0.933	0.001	—	—	—
MSE salts (SG6)	5.921 {8.7e4}	45.996	0.667[b]	—	—	—
MSE salts (SG7)	4.399 {7.8e4}	31.902	0.550[c]	—	—	—
Glass (SG8)	0.541 {5,578}	6.527	0.007	—	—	—
Raschig rings (SG9)	0.080 {1,200}	0.961	0.001	—	—	—
Mixed metals (SG10)	1.046 {1.8e4}	3.282	0.230[d]	—	—	—
Zero matrix (SG11)	0.042	0.080	0.011	—	—	—

a. 78% ^{241}Am mass not traceable.
b. 34% ^{241}Am mass not traceable.
c. 41% ^{241}Am mass not traceable.
d. 98% ^{241}Am mass not traceable.

Table 4. Actual waste test sample best estimate radioactive material loadings.

Actual Rocky Flats Test Samples	Total TRU Activity (Ci) {α nCi/g}	^{239}Pu Mass (g)	^{240}Pu Mass (g)	^{241}Am Mass (g)	^{235}U Mass (g)	^{238}U Mass (g)
Graphite (RF1)	0.502 {1.4e4}	5.879	0.367	0.011	—	—
Graphite (RF2)	0.799 {1.2e4}	9.270	0.579	0.020	—	—
Combustibles (RF3)	0.431 {1.3e4}	4.877	0.305	0.014	0.542	—
Combustibles (RF4)	0.002 {56}	0.020	0.001	6.240e-05	0.006	—
Filters/insulation (RF5)	0.285 {4,825}	3.295	0.206	0.007	0.015	—
Filters/insulation (RF6)	11.998 {4.0e5}	143.687	8.090	0.249	—	—
Inorganic sludge (RF7)[a]	2.564 {1.7e4}	0.572	0.036	0.735	0.846	—
Inorganic sludge (RF7)[b]	1.790 {1.2e4}	0.481	0.030	0.511	0.846	—
Inorganic sludge (RF8)[a]	0.353 {1,782}	2.348	0.147	0.049	0.633	295.358
Inorganic sludge (RF8)[b]	0.364 {1,838}	2.650	0.166	0.046	0.633	—
Inorganic sludge (RF9)[a]	0.949 {6,683}	1.050	0.691	0.022	—	—
Organic sludge (RF10)[a]	0.085 {607}	0.980	0.061	0.002	0.018	—
Organic sludge (RF11)[a]	0.148 {643}	1.712	0.107	0.004	0.018	—
Organic sludge (RF11)[b]	0.114 {496}	1.350	0.085	0.002*	0.177	—
MSE (RF12)	15.019 {4.8e5}	161.332	9.612	0.686	—	—
MSE (RF13)	73.765 {9.9e5}	277.731	17.355	15.126	2.086	—
Glass (RF14)	0.182 {2,141}	2.027	0.127	0.006	0.450	—
Glass (RF15)	0.238 {4,091}	2.722	0.170	0.007	0.056	—

Table 4. (continued).

Actual Rocky Flats Test Samples	Total TRU Activity (Ci) {α nCi/g}	^{239}Pu Mass (g)	^{240}Pu Mass (g)	^{241}Am Mass (g)	^{235}U Mass (g)	^{238}U Mass (g)
Glass (RF16)	7.783 {2.0e5}	76.417	4.775	0.510	2.420	—
Raschig ring (RF17)	0.087 {1,323}	0.961	0.060	0.003	—	—
Raschig ring (RF18)	0.615 {1.3e4}	7.045	0.440	0.017	—	—
Mixed metals (RF19)[c]	— {—}	—	—	—	—	—
Mixed metals (RF20)	0.437 {3,624}	3.969	0.248	0.036	0.099	190.000
Mixed metals (RF21)	1.575 {2.4e4}	17.968	1.123	0.047	—	—

a. SAS active mass measure basis.
b. Radiochemistry data basis.
c. Blank sample, no activity.

3.3 Capability Targeting Test Samples

Nondestructive waste assay systems must possess a number of capabilities. These capabilities are related to the ability to adequately segregate at 100 nCi/g TRU alpha activity, appropriately establish the correct radionuclidic/isotopic composition, and accommodate waste form characteristics known to complicate existing technologies, e.g., high uncorrelated neutron component, high density matrices, etc. Several test samples exhibiting these characteristics are indicated to aid the evaluator in the assessment of these capabilities. The attributes and complicating factors of these particular test samples are discussed individually.

For simplicity of discussion, the capability targeting test samples are grouped under the general headings of: segregation at 100 nCi/g, radionuclidic/isotopic composition identification, and general complicating configurations. It is not intended that these categories be all inclusive of waste NDA challenges, rather they serve as indicators of the capability for some attributes that should be accommodated by a proficient and comprehensive waste NDA technology.

3.3.1 Segregation at 100 nCi/g

There are two test samples within this category: graphite surrogate, SG1, and combustibles actual waste test sample, RF4. The graphite surrogate SG1 was specifically loaded with a uniform distribution of nominal weapons grade plutonium to yield an alpha activity concentration near the QAPP minimum detectable concentration criterion of 60 nCi/g. The actual alpha activity concentration of the SG1 test sample is 73 nCi/g. The graphite matrix is, by most standards, considered to present minimal interference to existing waste NDA technologies and therefore makes an excellent sample for evaluating sensitivity at the low-level/TRU segregation point.

The best estimate alpha activity concentration for actual waste test sample RF4 (moist combustibles) is 56 nCi/g. Similar to the graphite SG1 test sample, the combustibles matrix is not considered to present a significant matrix complication to existing waste NDA technologies. Although the RF4 test sample is convenient for consideration of segregation sensitivity, it also has a unique feature in that it contains a small quantity of ^{235}U, which also must be quantified.

3.3.2 Radionuclidic/Isotopic Composition Identification

The moist combustibles actual waste test sample RF3 is a good sample for evaluating the ability of a system to properly identify and quantify a radionuclidic composition other than standard weapons grade plutonium. This sample contains ^{235}U in addition to weapons grade plutonium at a weight fraction of approximately 12%. The mass of ^{235}U is in excess of 0.5 grams and should be readily detected. The matrix is combustibles, generally considered not to pose a significant complication to existing waste NDA technologies. Failure of a given system to, at a minimum, identify the presence of ^{235}U is indicative of poor or nonexistent ability to accommodate waste forms that have radionuclidic distributions other than that of typical weapons grade plutonium.

The glass actual waste test sample RF14 is similar to RF3 in that the sample contains an enhanced ^{235}U to weapons grade plutonium weight fraction in excess of 20%. The glass matrix, although more dense than combustibles, should be accommodated by most waste NDA techniques. This sample also provides an indication of a given technology to detect and quantify radionuclidic distributions other than that of standard weapons grade plutonium.

The surrogate test sample SG10 is a mixed metals matrix with an elevated ^{241}Am to weapons grade plutonium weight fraction of 6.6%. The volume averaged matrix density should not be outside the capability of existing waste NDA technologies. This sample also provides an opportunity to test the robustness of the plutonium isotopics determination in that excessive amounts of ^{241}Am can interfere with this process.

3.3.3 General Complicating Configurations

Test sample types included in the general complicating configurations are those that exhibit combinations of attributes that impact important capability functions. An example of such a test sample is one that possesses attributes that affect LLW/TRU segregation capability, the ability to correctly identify and quantify radionuclidic compositions in a matrix of high density.

Surrogate test sample SG11, the empty or zero matrix surrogate, was configured to yield information on measurement system bias obtained in the absence of matrix induced interferences. Additionally, the SG11 sample radioactive material composition was intended to present an interference since the ^{241}Am/weapons grade plutonium ratio is elevated with respect to nominal weapons grade plutonium at a mass ratio of 0.123. The SG11 sample alpha activity loading is equivalent to 100 nCi/g in 100 pounds of matrix material providing information on system sensitivity. The participant was required to quantitate the ^{241}Am mass within the range of ± 28% to yield an acceptable alpha activity per cent recovery (%R) provided the plutonium mass is correct.

The surrogate test sample SG4, inorganic sludge, contains attributes that may manifest as multiple complications to a waste NDA system. This test sample contains a matrix with a high density, approximately 1.1 g/cm^3, which is known to pose difficulty to most waste assay technologies. Secondly, the radionuclidic distribution departs significantly from the nominal weapons grade plutonium radionuclidic specification as follows: 0.154 g ^{239}Pu, 0.02g ^{241}Am, 8.2 g ^{235}U, 0.038 g ^{234}U, and 3.8 Kg ^{238}U. This relatively small quantity of ^{239}Pu complicates the determination of the radionuclidic composition because most determination routines use ^{239}Pu as the reference. The SG4 test sample represents a number of complications to the waste NDA systems, however, it is a reasonable representation of attributes associated with the inorganic sludge waste population.

The above capability targeting test samples were singled out to assist the end user in the assessment of performance. There are many other CEP test samples that present attributes that produce confounding effects on waste NDA systems. The test samples identified above will be revisited in the results discussion section with the intent of exposing deficiencies or confirming capability.

4. CEP EVALUATION PARAMETERS AND RESULTS

Primary performance evaluation parameters are total bias and relative precision. Minimum detectable concentration is addressed, but not analyzed in a rigorous manner. The bias and precision evaluation determinations are based on the techniques employed in the DOE-CAO PDP plan. These techniques are briefly discussed followed by the presentation of results per these parameters. Detailed information on the computation of the precision parameter, relative standard deviation, and the total bias limits on the two-sided 95% confidence bounds for the ratio of the mean of measured value to the known or accepted reference value are found in the CEP test plan.

It is important to note that performance on total bias and relative precision, within the context of the DOE-CAO requirements, is with reference to total TRU alpha activity. This value is tabulated and is the basis for determining acceptable or unacceptable performance with respect to DOE-CAO requirements. In addition to this assessment, results are indicated in terms of percent recovery on the mass of the various radionuclides contained in the test samples. This is significant in that much more information on the functionality and capability of a given waste NDA system can be obtained through data quoted in this form.

4.1 Total Bias

As defined for the project, bias is the systematic or constant component of the total error or total measurement uncertainty. Measurement system bias on total TRU alpha activity is assessed through the processing of eight replicate measures of a given test sample for the purpose of defining the mean of the bias distribution. Comparing measurement system reported mean total alpha activity values to the known or reference value allows determination of average percent recovery and the confidence interval endpoints for the true value at a 95% confidence level. The upper and lower 95% confidence limits for the two-sided Student's t distribution are used to modify the limits in Table 1, Columns 2 and 3, for performance assessment purposes. Satisfactory performance is indicated if the ratio of the mean measured TRU alpha activity value to the known or reference value, expressed as a percent, is within the computed upper and lower 95% confidence bounds.

This method of bias assessment and scoring per established QAOs is rigorous when the radioactive material composition and mass, μ, is precisely known. In the case of the actual waste type test sample, the reference value, μ, is not precisely known, but rather is a best estimate of the radioactive material loading with an uncertainty. As reported in the results section, the reference basis for bias scoring of actual waste test samples is derived from best estimate radionuclide content as determined by the INEEL SAS. For most of the actual waste test samples, the assigned radioactive material composition and associated mass values are reasonable and defensible. Although there is uncertainty associated with SAS based actual waste test sample reference values, it is not accounted for in the initial evaluation of bias as tabulated in the results section. For this reason, total bias results are referred to as "first pass" indicating that further refinement of the analysis is needed. A more comprehensive analysis accounting for the actual waste test sample reference value uncertainty will be presented in the Mixed Waste Focus Area/Characterization Monitoring Sensor Technology Nondestructive Waste Assay Capabilities Evaluation Program—Final Report.

4.2 Relative Precision

Precision is a measure of the random error component of the total error or total uncertainty sometimes called the repeatability or repeatability error. As defined for the project, relative precision is expressed as the ratio of the standard deviation, s, of the TRU alpha activity derived from eight replicate measurements of a test sample to the known or reference alpha activity value, μ, for the test sample. All test samples, surrogate and actual waste, will be evaluated per the interfering precision QAOs in Table 2. These QAOs are derived from the 95% upper confidence bound on the true system precision specified in the QAPP, Table 9-1. As indicated in the total bias section, the reference value used for the actual waste test samples contains uncertainty that is not accounted for in the "first pass" analysis. This will be addressed in the CEP final report.

4.3 Minimum Detectable Concentration Evaluation

The TRU alpha activity minimum detectable concentration (MDC) is dependent upon the count time and count rate, which can be dependent on the composition and configuration of the waste matrix and the amount and type of interfering radionuclides present. Measurement system MDCs are typically evaluated through the acquisition of replicate measurement data on a blank sample that contains matrix material or other radionuclides representing interferences in a configuration typical of actual wastes. Because of time constraints associated with the conduct of the project, no blank samples were prepared to support a strict MDC evaluation. Nevertheless, general statements regarding measurement system MDC can be made as a function of test sample configuration using the percent relative standard deviation (%RSD) determined from participant test sample reports. It is emphasized that the MDC information obtained in this manner pertains to the interferences manifested specific to the particular test sample matrix/source configuration. As with all counting situations, longer counting times in many instances will reduce the MDCs.

Criteria used to estimate MDC as a function of test sample configuration are listed below. The basis and derivation of these criteria will be documented in the CEP final report. These criteria should be used as approximate boundaries for determining whether the system MDC, specific to the test sample configuration, is either above or below the radioactive material loading of the sample:

- If the measured %RSD is less than 30%, it is probable that the MDC for that test sample configuration is less than the radioactive material loading.

- If the measured %RSD is greater than 36%, it is probable that the MDC for that test sample configuration is greater than the radioactive material loading.

The further the measured %RSD values are from these limits, the more confidence there is that the MDC is less than or greater than the configuration specific radioactive material loading. For values outside the range 15—54%, it is estimated that the confidence level is >95%. For test sample configurations in or close to the 30—36% range, the radioactive material loading is close to the MDC value. For interpretation purposes regarding the MDC parameter, (%RSD $\sigma/\bar{x}$ per Test Sample α Ci Configuration) Column 4 in the Precision/MDC tables is to be used to compare to the 30 and 36% limits discussed above.

4.4 CEP Test Results

Evaluation results for the total bias and relative precision parameters, in addition to MDC data, are presented in this section via a series of tables and plots. Total bias is presented in terms of percent recovery, %R, on the alpha activity and mass of select radionuclides. Tables of relative precision data are provided in terms of %RSD on total TRU alpha activity expressed as, $(\sigma/\mu) \times 100$, for comparison to DOE-CAO based QAOs and %RSD expressed as, $(\sigma/\bar{x}) \times 100$, for MDC evaluation purposes. The %RSD, $(\sigma/\bar{x}) \times 100$, obtained on the primary measured quantity mass, i.e., ^{239}Pu and ^{240}Pu, for each system is provided for information.

Analysis results are organized per participant where the hardware and software configurations used for data acquisition and analysis are specifically called out. It is important to note that these results apply only to the stated configuration. Suppliers of NDA characterization services continually refine their techniques. Therefore, it is important that the end user be cognizant of the hardware configuration and that software versions used to develop the results in this report may be different in the future or may have been modified to address a specific waste stream or inventory. Also note that results reported for the actual waste test samples are referenced to the optimized SAS measurements. These reference values have uncertainty in them that has not been accounted for in the following presentation. For all participants, the surrogate bias evaluation result table is presented first, the "first pass" actual waste test sample result table second, precision/MDC data third, and the average percent recovery by mass plots last.

Tabulated bias data directly related to the performance assessment mechanism implemented in the NDA PDP is the total alpha activity percent recovery in Column 2 of the Surrogate Bias Result tables. This parameter is tabulated for each surrogate test sample as identified in Column 2. Compliance with NDA PDP quality assurance objectives can be assessed for each test sample by examining the alpha activity average %R value relative to the Lower %R and Upper %R 95% confidence interval endpoints in Columns 3 and 4, respectively. Acceptable performance on this parameter is indicated if the alpha activity %R is within the range defined by the Lower %R and Upper %R endpoints. This same method of performance interpretation applies to the "First Pass" Bias Evaluation Results (Actual Waste Test Sample) tables with the exception that values cannot be considered as a direct indication of compliance with NDA PDP prescriptions due to the unaccounted for uncertainty in the reference value used to determine average alpha activity, %R.

Included in the bias evaluation result tables are percent recoveries by mass for ^{239}Pu, ^{240}Pu (Canberra HENC only), ^{241}Am, ^{235}U, and ^{238}U. These %R by mass values are intended to further elucidate system capability and/or limitation with respect to accurate quantitation of all radionuclides within a given test sample. In particular, they are included to point out that acceptable results on alpha activity %R do not always indicate a comprehensive capability. For example, a high measurement bias on ^{239}Pu mass that is in reality unacceptable can be offset by a low bias on the ^{241}Am mass such that the associated alpha activity %R falls within the acceptable range as specified by the NDA PDP scoring technique. It is therefore important that the end user fully consider all bias data presented such that a complete understanding of the assay system capability under consideration be appreciated.

Plots of average percent recovery by mass for select radionuclides are provided to allow ready visualization of system performance. The plots are percent recovery by mass versus test sample radionuclide mass ordered in ascending fashion. Because of the large range of radionuclide mass loadings over the test sample set, the abscissa (radionuclide mass) is in a log scale. Percent recovery data are included for all test samples, excepting those that exceed the 36% MDC criteria. It is of little value to show percent recovery data for a test sample that has been determined to be below the MDC of the system. Test samples exhibiting waste matrix/radionuclide configurations below the MDC of a particular assay system, i.e., greater than 36%, are identifiable in the Precision/MDC tables.

Precision results and MDC data are tabulated in the same table for each participant. Evaluation of compliance with NDA PDP QAOs for precision can be directly assessed for the surrogate type test samples. Column 2 of the Precision/MDC tables tabulates %RSD as ($\sigma/\mu \times 100$), where σ is the standard deviation of the reported alpha activity and μ is the known alpha activity value. Column 3 contains range specific, alpha activity precision QAOs. Acceptable performance is indicated if the %RSD determined from the reported alpha activity values is less than or equal to the Column 3 precision QAO. An indirect assessment of compliance with NDA PDP performance parameters can be made for the actual waste test samples in the same manner. Again it is termed indirect as the value μ has uncertainty associated with it.

For purposes of the MDC evaluation, %RSD as ($\sigma/\bar{x} \times 100$) is computed from the reported alpha activity, where σ is the standard deviation of the reported alpha activity and $\bar{x}$ is the average reported alpha activity value. This value is tabulated in Column 4 and can be interpreted per the discussion in the MDC section. The %RSD in terms of mass of the primary measured quantity, i.e., ^{239}Pu or ^{240}Pu, in units of ($\sigma/\bar{x} \times 100$), is provided in Column 5. This may be useful for the case where MDC has been defined in terms of mass. Also note the alpha activity loading and alpha activity concentration in units of nCi/g are tabulated for each test sample in Column 6.

4.4.1 Bio-Imaging Research (BIR)—Waste Inspection Tomography (WIT) System

The BIR WIT total bias results for all samples processed are shown in Table 5. The corresponding relative precision and MDC data are given in Table 6. Plots of average percent recovery relative to mass of radionuclide are given in Figures 1 and 2. Average percent recovery specific to ^{241}Am and ^{235}U as presented in the bias result table and the plots are not completely representative of the true WIT capability as no radionuclidic/isotopic analysis routines were implemented at the time of CEP testing. Because of constraints associated with BIR WIT data acquisition time, only a limited number of test samples were evaluated. The results in this section are based on measurements acquired by the WIT single detector hardware configuration in conjunction with the A&PCT, Revision 1.3, software package, copyright University of California. CEP evaluations and results apply to this declared BIR WIT hardware/software configuration.

Table 5. BIR WIT total bias results.

Surrogate Test Sample	Total Avg %R ($\bar{x}/\mu$)	Total α % Recovery Acceptance Criteria (95% Confidence Bounds)		^{239}Pu Mass Avg (%R)	^{241}Am Mass Avg (%R)	^{235}U Mass Avg (%R)	^{238}U Mass Avg (%R)
		Lower %R	Upper %R				
MSE salts (SG6)	70.7	50.9	149.1	103.5	15.4	—	—
Raschig rings (SG9)	154.9	33.5	196.5	146.0	284.4	—	—
Actual Rocky Flats Test Samples	**Total α Avg %R ($\bar{x}/\mu$)**	**Total α % Recovery Acceptance Criteria (95% Confidence Bounds)**		**^{239}Pu Avg (%R)**	**^{241}Am Avg (%R)**	**^{235}U Avg (%R)**	**^{238}U Avg (%R)**
		Lower % R	**Upper %R**				
Organic sludge (RF11)[a]	161.4	35.0	195.0	144.9	141.9	0.0	—
Organic sludge (RF11)[b]	191.0	35.9	194.1	183.8	242.9	0.0	—
Metals (RF20)	96.8	30.7	199.3	121.2	28.9	0.0	0.0

a. SAS active mass evaluation basis.
b. Radiochemistry evaluation basis.

Table 6. BIR WIT relative precision/MDC data.

Test Sample	%RSD [(s/μ) x 100] per Test Sample TRU α (Ci) Configuration	Precision QAO %RSD on α (Ci)	%RSD [($\bar{s}/\bar{x}$) x 100] per Test Sample TRU α (Ci) Configuration	%RSD [($s/\bar{\bar{x}}$) x 100] per Test Sample ^{239}Pu Mass	Test Sample Total α activity (Ci) {nCi/g}
RF11 (organic sludge)	5.0[a] 5.9[b]	<14.0	3.1	3.1	0.148[a] {643} 0.114[b] {496}
SG6 (MSE salts)	1.1	<7.0	1.5	1.5	5.921 {8.7E4}
SG9 (raschig ring)	4.2	<14.0	2.7	2.7	0.07955 {1.2E3}
RF20 (mixed metals)	0.8	<14.0	0.8	0.8	0.4367 {3.6E3}

a. SAS active mode data basis.
b. Radiochemistry data basis.

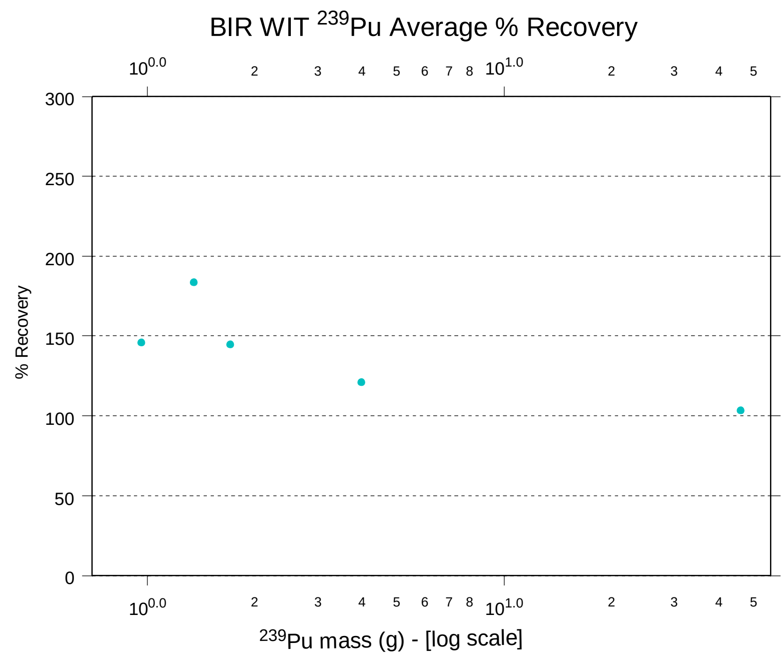

Figure 1. Plot of %Recovery as a function of ^{239}Pu mass for the Bio-imaging Research WIT System.

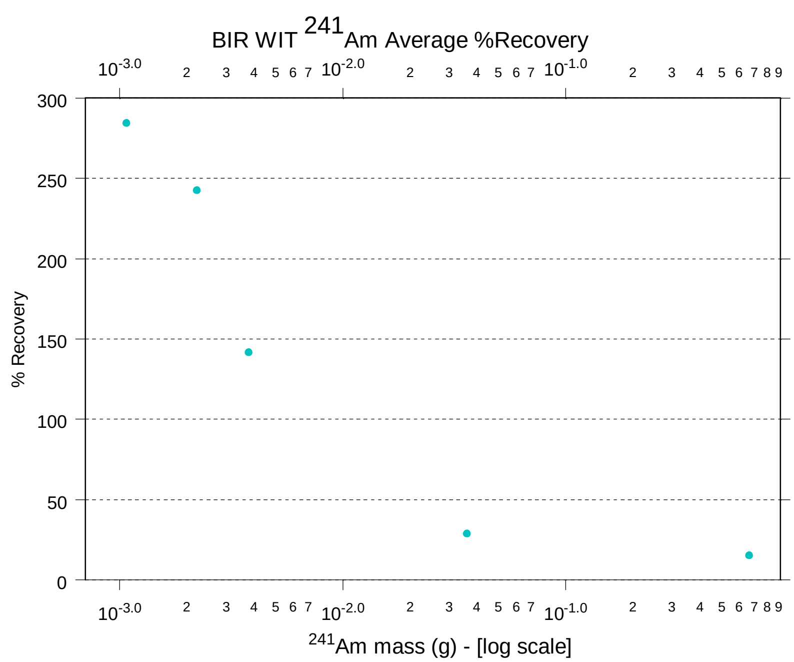

Figure 2. Plot of %Recovery as a function of ^{241}Am mass results for the Bio-Imaging Research WIT System.

4.4.2 Canberra Industries, Inc.—Segmented Gamma Scanner (SGS)

The SGS total bias results for the surrogate test samples are presented in Table 7, and "first pass" bias results for actual test samples are presented in Table 8. Relative precision and MDC data for all test samples grouped according to waste form characteristic are given in Table 9. Figures 3, 4, and 5 present average percent recovery as a function of radionuclide mass. CEP test results are based on measurements acquired by the Canberra WM2210T SGS system using the Canberra Gamma Waste Assay Software, Version 2.2, application package in conjunction with the MGA V9.5a isotopics software package and the MGAU uranium isotopics software package. Supportive data processing and reduction is performed using the Canberra SGS_REV4.xls spreadsheet for differential peak correction and determination of derived quantities such as total alpha activity. All CEP evaluations and results apply to this Canberra declared SGS hardware/software configuration.

Table 7. Canberra Industries SGS total bias results (surrogate test samples).

Surrogate Test Samples	Total α Avg %R ($\bar{\bar{x}}/\mu$)	Total α % Recovery Acceptance Criteria (95% Confidence Bounds) Lower % R	Upper % R	^{239}Pu Avg (%R)	^{241}Am Avg (%R)	^{235}U Avg (%R)	^{238}U Avg (%R)
Graphite (SG1)	649.3	160.7	54.3	117.6	11,888.0	—	—
Combustibles (SG2)	89.2	33.3	196.7	87.8	90.6	—	—
Filters/insul (SG3)	116.2	51.6	148.4	121.1	91.6	—	—
Inorganic sludge (SG4)	145.2	81.1	148.9	129.5	313.8	3.5	60.3
Organic sludge (SG5)	51.5	35.1	194.9	48.4	49.5	—	—
MSE salts (SG6)	148.3	67.5	132.5	125.1	176.6	—	—
MSR salts (SG7)	143.0	59.7	140.3	162.1	106.5	—	—
Glass (SG8)	93.2	32.4	197.6	91.6	95.6	—	—
Raschig rings (SG9)	61.1	36.2	193.8	61.5	66.4	—	—
Metals (SG10)	62.5	32.1	197.9	72.4	58.8	—	—
Empty (SG11)	164.2	78.1	121.9	115.9	166.5	—	—

Table 8. Canberra Industries SGS first pass bias evaluation results (actual waste test samples).

Actual Rocky Flats Test Samples	Total α Avg %R ($\bar{x}/\mu$)	Total α % Recovery Acceptance Criteria (95% Confidence Bounds) Lower % R	Upper % R	^{239}Pu Avg (%R)	^{241}Am Avg (%R)	^{235}U Avg (%R)	^{238}U Avg (%R)
Graphite (RF1)	119.9	38.4	191.6	117.1	146.2	—	—
Graphite (RF2)	108.3	33.2	196.8	107.4	111.4	—	—
Combustibles (RF3)	70.5	31.5	198.5	70.9	66.0	44.8	—
Combustibles (RF4)	7.2	40.1	174.9	0.0	4.8	0.0	—
Filters (RF5)	169.9	41.2	188.8	163.2	177.1	0.0	—
Filters (RF6)	37.1	51.5	148.5	36.3	32.9	—	—
Inorganic sludge (RF7)[a]	43.6	65.4	134.6	0.0	43.8	0.0	0.0
Inorganic sludge (RF7)[b]	52.0	62.5	184.6	0.0	63.1	0.0	—
Inorganic sludge (RF8)[a]	93.3	35.7	194.3	93.6	91.2	61.3	83.8
Inorganic sludge (RF8)[b]	90.6	35.6	194.4	82.9	96.5	61.3	—
Inorganic sludge (RF9)[a]	64.9	30.9	199.1	64.9	57.8	—	—
Organic sludge (RF10)[a]	60.3	34.9	195.1	58.7	56.9	0.0	—
Organic sludge (RF11)[a]	133.7	38.2	191.8	102.4	440.4	0.0	—
Organic sludge (RF11)[b]	73.4	40.6	189.4	129.9	754.0	0.0	—
MSE (RF12)	138.2	61.5	138.5	140.9	131.3	—	—
MSE (RF13)	29.1	50.6	149.4	19.1	31.5	0.0	—
Glass (RF14)	105.3	33.2	196.8	104.4	90.2	89.3	—
Glass (RF15)	83.1	34.9	195.1	82.2	81.7	0.0	—
Glass (RF16)	24.3	41.5	148.5	24.6	23.6	20.0	—
Raschig ring (RF17)	82.1	32.8	197.2	85.2	61.5	—	—
Raschig ring (RF18)	83.0	32.4	197.6	82.4	84.9	—	—
Metals (RF19)[c]							
Metals (RF20)	80.8	34.6	195.4	92.3	51.2	95.0	2,730
Metals (RF21)	75.9	31.9	198.1	76.2	70.1	—	—

a. SAS active mass basis.
b. Radiochemistry data basis.
c. Blank test sample, no detectable activity.

Table 9. Canberra Industries SGS relative precision/MDC data.

Test Sample	%RSD [(s/μ) x 100)] per Test Sample TRU α (Ci) Configuration	Precision QAO % RSD on α (Ci)	%RSD [(s/$\bar{x}$) x 100] per Test Sample TRU α (Ci) Configuration	% RSD [(s/$\bar{x}$) x 100)] per Test Sample ^{239}Pu (g) Measure	Test Sample Total α activity (Ci) {nCi/g}
RF1 (graphite)	10.0	<14.0	8.4	7.9	0.5029 {1.4E4}
RF2 (graphite)	3.8	<14.0	3.5	3.3	0.7986 {1.2E4}
SG1 (graphite)	143.7	<18.0	22.1	28.5	0.0071 {73}
RF3 (dry combustibles)	1.8	<14.0	2.5	2.4	0.4313 {1.3E4}
RF4 (dry combustibles)	0.1	<18.0	1.6	1.7	0.0018 {56}
SG2 (dry combustibles)	4.0	<14.0	4.4	4.5	0.3144 {7,149}
RF5 (filters/ insulation)	13.3	<14.0	7.8	3.6	0.2847 {4,825}
RF6 (filters/ insulation)	1.7	<7.0	4.7	4.7	11.998 {4.0E5}
SG3 (filters/ insulation)	2.0	<7.0	1.7	1.6	4.891 {1.22E5}
RF7 (inorganic sludge)	18.3[a] 26.2[b]	<7.0 <14.0	41.9	42.0	2.564[a] {1.7E4} 1.79[r] {1.2E4}
RF8 (inorganic sludge)	6.8[a] 6.6[b]	<14.0	7.3	6.8	0.353[a] {1782} 0.364[b] {1,838}
SG4 (inorganic sludge)	60.8	<14.0	41.9	40.5	0.0836 {694}
RF9 (inorganic sludge)	1.1[a]	<14.0	1.7	1.6	0.949[a] {6,683}
RF10 (organic sludge)	5.9[a]	<14.0	9.8	6.4	0.085[a] {607}
RF11 (organic sludge)	9.7[a] 12.6[b]	<14.0	7.3	6.2	0.148[a] {643} 0.114[b] {496}
SG5 (organic sludge)	6.1	<14.0	11.9	13.0	0.0774 {508}

Table 9. (continued).

Test Sample	%RSD [(s/μ) x 100)] per Test Sample TRU α (Ci) Configuration	Precision QAO % RSD on α (Ci)	%RSD [(s/$\bar{x}$) x 100] per Test Sample TRU α (Ci) Configuration	% RSD [(s/$\bar{x}$) x 100)] per Test Sample ^{239}Pu (g) Measure	Test Sample Total α activity (Ci) {nCi/g}
RF12 (MSE salts)	13.7	<7.0	9.9	10.0	15.019 {4.8E5}
RF13 (MSE salts)	0.7	<7.0	1.8	2.3	73.765 {9.9E5}
SG6 (MSE salts)	20.8	<7.0	14.0	13.9	5.921 {8.7E4}
SG7 (MSE salts)	11.5	<7.0	8.0	10.7	4.399 {7.8E4}
RF14 (glass)	3.8	<14.0	3.6	2.7	0.182 {2,141}
RF15 (glass)	5.8	<14.0	7.0	6.5	0.238 {4,091}
RF16 (glass)	1.8	<7.0	7.6	7.5	7.783 {2.0E5}
SG8 (glass)	2.8	<14.0	3.1	3.0	0.5412 {5,578}
RF17 (raschig ring)	3.4	<14.0	4.1	4.2	0.0872 {1,323}
RF18 (raschig ring)	2.8	<14.0	3.4	3.4	0.6153 {1.3E4}
SG9 (raschig ring)	7.3	<14.0	12.0	12.0	0.0796 {1.2E3}
RF19 (mixed metals)			8.5	8.6	0.0
RF20 (mixed metals)	6.8	<14.0	6.8	6.4	0.4367 {3624}
RF21 (mixed metals)	2.3	<14.0	3.0	2.8	1.5746 {2.4E4}
SG10 (mixed metals)	4.0	<14.0	4.0	6.4	1.0464 {1.8E4}
SG11 (zero matrix)	33.5	<14.0	20.4	27.5	0.0422 {1.8E4}

a. SAS active mode basis.
b. Radiochemistry data basis.

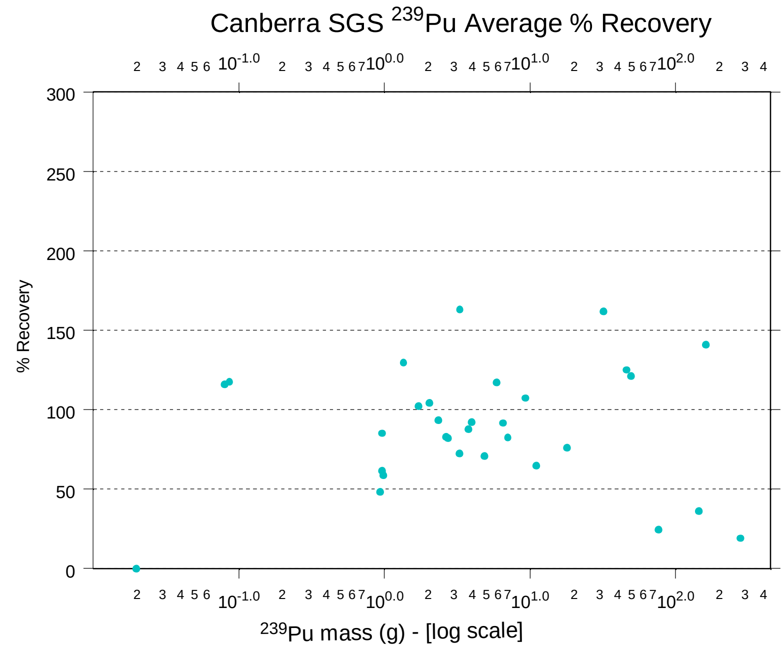

Figure 3. Plot of % Recovery as a function of ^{239}Pu mass for the Canberra Industries SGS System.

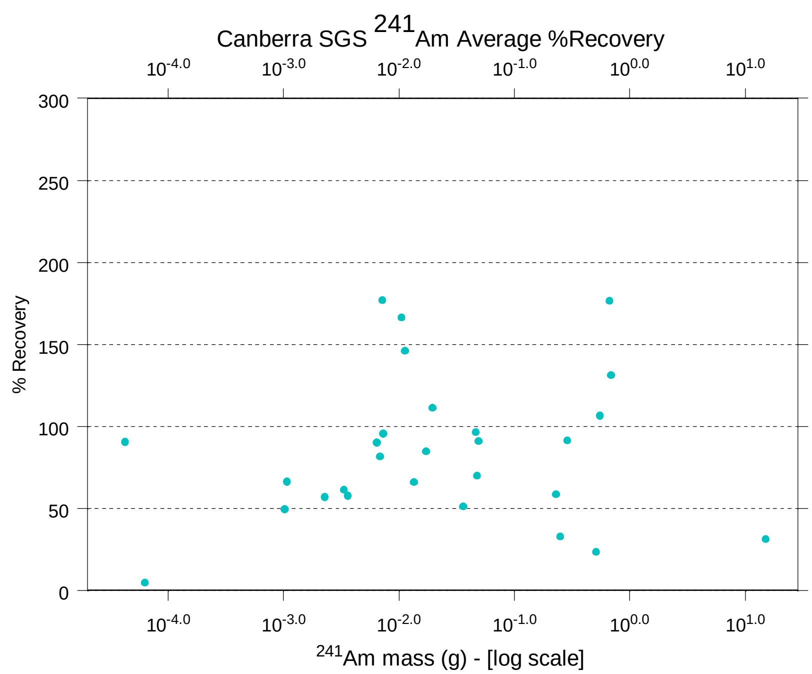

Figure 4. Plot of % Recovery as a function of ^{241}Am mass for the Canberra Industries SGS System.

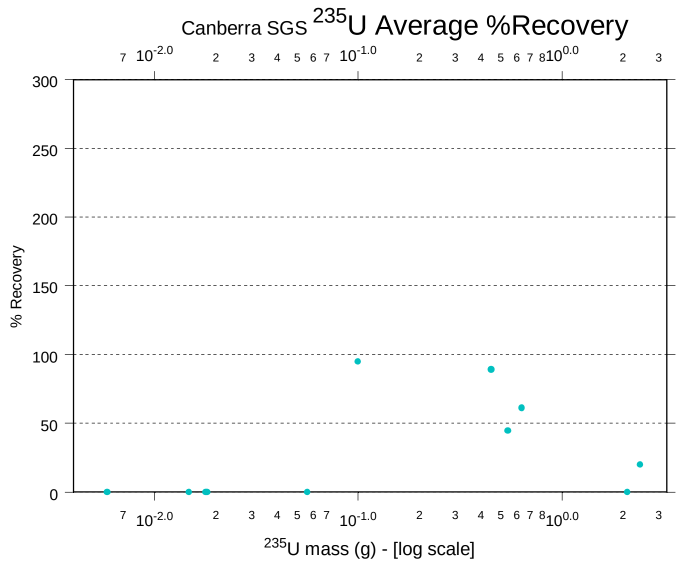

Figure 5. Plot of % Recovery as a function of ^{235}U mass for the Canberra Industries SGS System.

4.4.3 Canberra Industries, Inc. High Efficiency Neutron Counter (HENC)

HENC total bias results for the surrogate and actual test samples are detailed in Tables 10 and 11, respectively. Table 12 provides the corresponding relative precision and MDC data for the HENC system. Figures 6, 7, and 8 give plots of average percent recovery as a function of radionuclide mass. CEP test results are based on measurements acquired by the Canberra HENC system using the Canberra Neutron Assay Software (NAS), Version 2.0A package in conjunction with the MGA V9.5a isotopics software package and the MGAU uranium isotopics software package. All CEP project evaluations and results apply to this Canberra declared HENC hardware/software configuration.

Table 10. Canberra Industries HENC total bias results (surrogate test samples).

Surrogate Test Samples	Total α Avg %R ($\bar{\bar{x}}/\mu$)	Total α % Recovery Acceptance Criteria (95% Confidence Bounds)		^{240}Pu Avg (%R)	^{241}Am Avg (%R)	^{235}U Avg (%R)	^{238}U Avg (%R)
		Lower % R	Upper % R				
Graphite (SG1)	173.4	50.6	164.4	141.7	193.0	—	—
Combustibles (SG2)	108.2	33.0	197.0	107.5	117.0	—	—
Filters/insul (SG3)	95.8	53.8	146.2	106.0	46.4	—	—
Inorganic sludge (SG4)	21.9	46.0	184.0	31.6	19.5	0.5	53.0
Organic sludge (SG5)	65.4	36.3	193.7	68.7	62.0	—	—
MSE salts (SG6)	123.9	70.8	129.2	114.6	156.4	—	—
MSR salts (SG7)	99.5	59.6	140.4	109.5	70.5	—	—
Glass (SG8)	99.6	32.6	197.4	98.3	107.0	—	—
Raschig rings (SG9)	110.4	36.8	193.2	102.2	120.7	—	—
Metals (SG10)	99.9	39.5	190.5	153.7	82.4	—	—
Empty (SG11)	146.3	103.8	96.2	190.1	148	—	—

Table 11. Canberra Industries HENC first pass bias results (actual waste test samples).

Actual Rocky Flats Test Samples	Total α Avg %R ($\bar{\bar{x}}/\mu$)	Total α % Recovery Acceptance Criteria (95% Confidence Bounds) Lower % R	Upper %R	^{240}Pu Avg (%R)	^{241}Am Avg (%R)	^{235}U Avg (%R)	^{238}U Avg (%R)
Graphite (RF1)	122.6	33.2	196.8	120.6	153.0	—	—
Graphite (RF2)	124.7	32.2	197.8	123.3	132.2	—	—
Combustibles (RF3)	89.2	34.5	195.5	88.8	80.7	61.8	—
Combustibles (RF4)	252.8	81.9	133.1	246.1	271.8	137.0	—
Filters (RF5)	137.0	42.3	187.7	136.2	156.8	0.0	—
Filters (RF6)	68.9	54.1	195.9	74.6	55.9	—	—
Inorganic sludge (RF7)[a]	2.4	52.5	147.5	135.9	0.001	0.0	0.0
Inorganic sludge (RF7)[b]	3.4	33.6	196.4	161.2	0.001	0.0	—
Inorganic sludge (RF8)[b]	65.3	34.0	196.1	58.1	70.4	40.9	—
Inorganic sludge (RF8)[a]	67.3	34.1	195.9	65.6	66.6	41.0	114.0
Inorganic sludge (RF9)[a]	121.2	38.3	191.7	120.3	112.2	—	—
Inorganic sludge (RF9)[b]							
Organic sludge (RF10)[a]	63.3	38.7	191.3	64.9	62.9	0.0	—
Organic sludge (RF10)[b]							
Organic sludge (RF11)[b]	128.0	38.0	192.0	119.5	154.9	0.0	—
Organic sludge (RF11)[a]	98.7	36.2	193.8	94.9	90.5	0.0	—
MSE (RF12)	114.5	55.2	144.8	110.8	96.2	—	—
MSE (RF13)	52.9	65.8	134.3	57.6	48.1	0.0	—
Glass (RF14)	110.0	31.7	198.3	113.6	106.7	0.0	—
Glass (RF15)	81.3	34.1	195.1	81.6	83.1	39.4	—

Table 11. (continued).

Actual Rocky Flats Test Samples	Total α Avg %R ($\bar{\bar{x}}/\mu$)	Total α % Recovery Acceptance Criteria (95% Confidence Bounds) Lower % R	Upper %R	^{240}Pu Avg (%R)	^{241}Am Avg (%R)	^{235}U Avg (%R)	^{238}U Avg (%R)
Glass (RF16)	58.3	45.7	149.5	59.3	53.1	33.3	—
Raschig ring (RF17)	79.0	35.2	194.8	79.7	59.1	—	—
Raschig ring (RF18)	78.9	32.5	197.5	78.0	81.2	—	—
Metals (RF19)[c]							
Metals (RF20)	108.0	37.9	192.1	102.4	101.0	97.0	1,814.9
Metals (RF21)	110.1	33.6	196.4	108.3	104.8	—	—

a. SAS active mode basis.
b. Radiochemistry data basis.
c. Blank, no detectable activity.

Table 12. Canberra Industries HENC relative precision/MDC data.

Test Sample	%RSD [(s/μ) 100)] per Test Sample TRU α (Ci) Configuration	Precision QAO %RSD on a (Ci)	%RSD [(s/$\overline{x}$) x 100)] per Test Sample TRU α (Ci) Configuration	%RSD [(s/$\overline{x}$ x 100)] per Test Sample ^{240}Pu mass	Test Sample Total α activity (Ci) {nCi/g}
RF1 (graphite)	3.8	<14.0	3.1	2.7	0.503 {1.4E4}
RF2 (graphite)	2.6	<14.0	2.1	1.0	0.799 {1.2E4}
SG1 (graphite)	12.6	<18.0	7.3	8.9	0.007 {73}
RF3 (dry combustibles)	5.4	<14.0	6.0	3.4	0.431 {1.3E4}
RF4 (dry combustibles)	49.8	<18.0	19.7	19.7	0.002 {56}
SG2 (dry combustibles)	3.5	<14.0	3.3	3.3	0.314 {7149}
RF5 (filters/insulation)	14.6	<14.0	10.7	1.4	0.285 {4825}
RF6 (filters/insulation)	4.9	<7.0	7.1	7.2	11.99 {4.0E5}
SG3 (filters/insulation)	4.6	<7.0	4.8	3.4	4.891 {1.22E5}
RF7 (inorganic sludge)	3.0[a] 4.3[b]	<14.0	126.7	126.6	2.564[a] {1.7E4} 1.79[b] {1.2E4}
RF8 (inorganic sludge)	4.9[a] 4.7[b]	<14.0	7.2	4.7	0.353[a] {1782} 0.364[b] {1838}
SG4 (inorganic sludge)	19.1	<14.0	87.0	93.5	0.084 {694}
RF9 (inorganic sludge)	10.8[a]	<14.0	8.2	7.2	0.949[a] {6683}
RF10 (organic sludge)	10.3[a]	<14.0	16.3	13.2	0.085[a] {607}
RF11 (organic sludge)	7.3[a] 9.5[b]	<14.0	7.4	5.8	0.148[a] {643} 0.114[b] {496}
SG5 (organic sludge)	7.5	<14.0	11.5	12.1	0.077 {508}
RF12 (MSE salts)	6.2	<7.0	5.4	1.4	15.02 {4.8E5}
RF13 (MSE salts)	18.8	<7.0	35.4	7.1	73.77 {9.9E5}
SG6 (MSE salts)	24.7	<7.0	20.0	28.6	5.921 {8.7E4}
SG7 (MSE salts)	11.4	<7.0	11.5	9.8	4.399 {7.8E4}
RF14 (glass)	2.0	<14.0	1.8	1.8	0.182 {2141}
RF15 (glass)	4.9	<14.0	6.0	3.3	0.238 {4,091}
RF16 (glass)	6.8	<7.0	11.7	12.6	7.783 {2.0E5}
SG8 (glass)	3.1	<14.0	3.1	2.1	0.541 {5,578}
RF17 (raschig ring)	6.2	<14.0	7.9	6.7	0.087 {1,323}
RF18 (raschig ring)	3.0	<14.0	3.8	3.1	0.615 {1.3E4}

Table 12. (continued)

Test Sample	%RSD [(s/μ) 100)] per Test Sample TRU α (Ci) Configuration	Precision QAO %RSD on a (Ci)	%RSD [(s/$\bar{x}$) x 100)] per Test Sample TRU α (Ci) Configuration	%RSD [(s/$\bar{x}$ x 100)] per Test Sample ^{240}Pu mass	Test Sample Total α activity (Ci) {nCi/g}
SG9 (raschig ring)	8.1	<14.0	7.3	1.8	0.0796 {1.2E3}
RF19 (mixed metals)		Blank	107.7	107.7	0
RF20 (mixed metals)	9.5	<14.0	8.8	3.0	0.437 {3,624}
RF21 (mixed metals)	4.3	<14.0	3.9	3.6	1.575 {2.4E4}
SG10 (mixed metals)	11.3	<14.0	11.3	11.2	1.0464 {1.8E4}
SG11 (zero matrix)	64.1	<14.0	43.8	60.2	0.0422

a. SAS active mode basis.
b. Radiochemistry data basis.

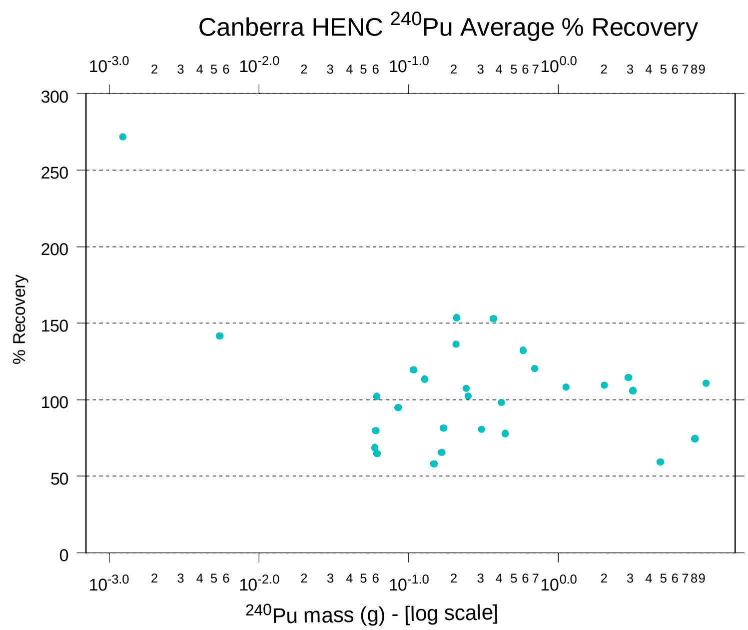

Figure 6. Plot of % Recovery as a function of ^{240}Pu mass for the Canberra Industries HENC System.

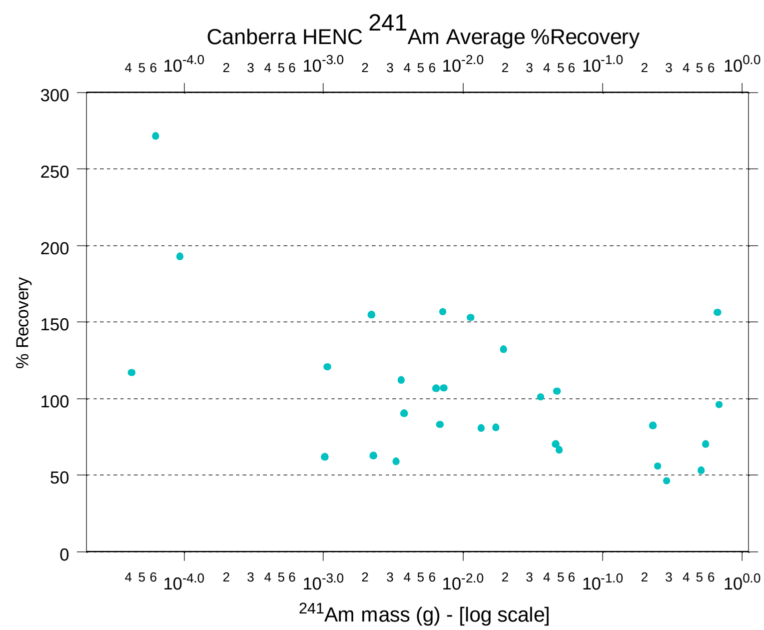

Figure 7. Plot of % Recovery as a function of ^{241}Am mass for the Canberra Industries HENC System.

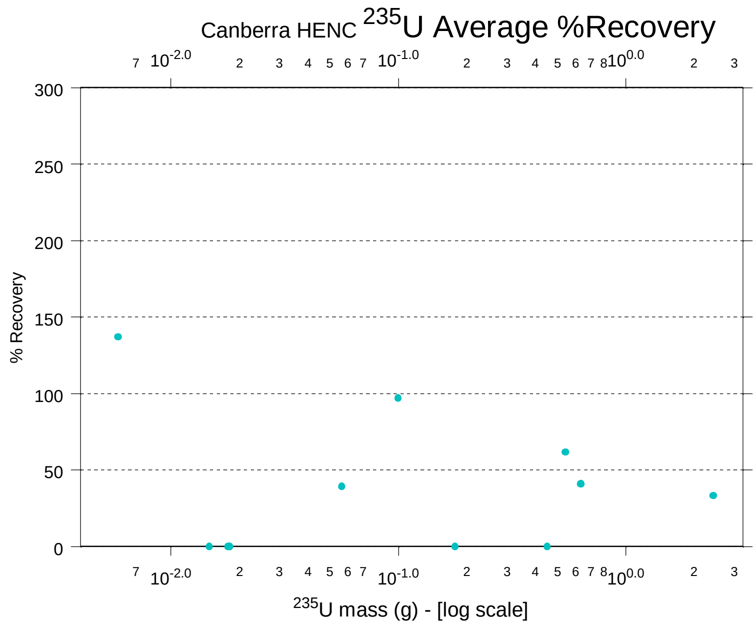

Figure 8. Plot of % Recovery as a function of ^{235}U mass for the Canberra Industries HENC System.

4.4.4 Canberra Industries - IQ3 Gamma Assay System

Tables 13 and 14 present the IQ3 bias results for the surrogate and actual waste test samples. Table 15 gives the corresponding precision/MDC data for the IQ3 System. Plots of average percent recovery of specific radionuclide are given in Figures 9, 10, and 11. The preliminary CEP test results tabulated below are based on measurements acquired by the Canberra IQ3 mobile assay system using the Genie-PC Waste Assay Software (Version 2.1) in conjunction with the MGA V9.5 isotopics and MGAU uranium isotopics software packages. Canberra IQ3 reports generated for CEP test samples are reduced using the IQ3_Rev. 3, TMU Report excel spreadsheet. All CEP evaluations and results for the Canberra IQ3 mobile assay system apply to this declared hardware/software configuration.

Table 13. Canberra Industries IQ3 bias results (surrogate test samples).

Surrogate Test Samples	Total α Avg %R ($\bar{x}/\mu$)	Total α % Recovery Acceptance Criteria (95% Confidence Bounds)		^{239}Pu Avg (%R)	^{241}Am Avg (%R)	^{235}U Avg (%R)	^{238}U Avg (%R)
		Lower % R	Upper %R				
Graphite (SG1)	104.3	45.8	169.2	107.7	117.2	—	—
Combustibles (SG2)	101.9	30.8	199.2	99.6	110.1	—	—
Filters/insul (SG3)	233.1	55.5	144.5	257.8	112.3	—	—
Inorganic sludge (SG4)	117.5	32.5	197.5	170.8	110.2	2.87	53.0
Organic sludge (SG5)	60.1	33.4	196.6	56.5	48.3	—	—
MSE salts (SG6)	68.3	61.2	138.8	53.1	86.2	—	—
MSR salts (SG7)	90.8	51.8	148.2	112.9	64.3	—	—
Glass (SG8)	109.8	31.2	198.8	106.4	118.1	—	—
Raschig rings (SG9)	91.2	32.2	197.8	91.7	100.5	—	—
Metals (SG10)	55.5	30.6	199.4	80.9	45.6	—	—
Empty (SG11)	109.8	54.8	145.2	124.0	100.6	—	—

Table 14. Canberra Industries IQ3 first pass bias results (actual waste test samples).

Actual Rocky Flats Test Samples	Total α Avg %R ($\overline{x}/\mu$)	Total α % Recovery Acceptance Criteria (95% Confidence Bounds) Lower % R	Upper % R	^{239}Pu Avg (%R)	^{241}Am Avg (%R)	^{235}U Avg (%R)	^{238}U Avg (%R)
Graphite (RF1)	113.8	32.7	197.3	109.8	142.1	—	—
Graphite (RF2)	110.3	30.9	199.1	108.8	117.0	—	—
Combustibles (RF3)	95.3	32.1	197.9	96.2	86.3	66.1	—
Combustibles (RF4)	165.4	58.4	156.6	164.8	187.3	93.0	—
Filters (RF5)	154.3	37.4	192.6	149.1	179.5	0.0	—
Filters (RF6)							
Inorganic sludge (RF7)[a]							
Inorganic sludge (RF7)[b]							
Inorganic sludge (RF8)[a]	63.9	36.5	193.5	60.6	60.9	37.7	114.0
Inorganic sludge (RF8)[b]	62.0	36.3	193.7	53.7	64.4	37.7	—
Inorganic sludge (RF9)[a]	63.4	32.3	197.7	63.2	58.7	—	—
Inorganic sludge (RF9)[b]							
Organic sludge (RF10)[a]	70.8	33.8	196.2	69.5	70.7	0.0	—
Organic sludge (RF10)[b]							
Organic sludge (RF11)[a]	92.2	33.2	196.8	91.6	84.6	0.0	—
Organic sludge (RF11)[b]	119.5	34.1	195.9	116.1	144.8	0.0	—
MSE (RF12)							
MSE (RF13)	30.6	52.3	147.7	45.3	2.2	0.0	—
Glass (RF14)	88.7	35.4	194.6	87.5	85.4	74.6	—
Glass (RF15)	87.6	31.1	198.9	86.2	89.8	170.2	—
Glass (RF16)							

Table 14. (continued).

Actual Rocky Flats Test Samples	Total α Avg %R ($\bar{x}/\mu$)	Total α % Recovery Acceptance Criteria (95% Confidence Bounds) Lower % R	Upper % R	^{239}Pu Avg (%R)	^{241}Am Avg (%R)	^{235}U Avg (%R)	^{238}U Avg (%R)
Raschig ring (RF17)	86.7	31.6	198.4	90.0	65.1	—	—
Raschig ring (RF18)	112.6	31.5	198.5	111.9	115.8	—	—
Metals (RF19)[c]							
Metals (RF20)	96.2	32.1	197.9	99.2	90.2	87.3	1,814.9
Metals (RF21)	97.7	36.4	193.6	96.6	93.1	—	—

a. SAS active mode basis.
b. Radiochemistry data basis.
c. Blank, no detectable activity.

Table 15. Canberra Industries IQ3 relative precision/MDC data.

Test Sample	%RSD [(s/μ) x 100] per Test Sample TRU α (Ci) Configuration	Precision QAO % RSD on a (Ci)	%RSD [(s/$\bar{x}$) x 100] per Test Sample TRU α (Ci) Configuration	%RSD [(s/$\bar{x}$ x 100)] per Test Sample ^{239}Pu Mass	Test Sample Total α Activity (Ci) {nCi/g}
RF1 (graphite)	3.2	<14.0	2.9	2.9	0.503 {1.4E4}
RF2 (graphite)	1.1	<14.0	1.0	1.0	0.799 {1.2E4}
SG1 (graphite)	6.9	<18.0	6.6	5.4	0.007 {73}
RF3 (dry combustibles)	2.6	<14.0	2.7	1.9	0.431 {1.3E4}
RF4 (dry combustibles)	21.9	<18.0	13.2	13.9	0.002 {56}
SG2 (dry combustibles)	1.0	<14.0	0.9	0.8	0.314 {7,149}
RF5 (filters/insulation)	8.8	<14.0	5.7	2.0	0.285 {4,825}
RF6 (filters/insulation)		<7.0			11.99 {4.0E5}
SG3 (filters/insulation)	6.5	<7.0	2.8	3.1	4.891 {1.22E5}
RF7 (inorganic sludge)		<14.0			2.564[a] {1.7E4} 1.79[b] {1.2E4}
RF8 (inorganic sludge)	7.8[a] 7.5[b]	<14.0	12.1	4.1	0.353[a] {1782} 0.364[b] {1,838}
SG4 (inorganic sludge)	3.0	<14.0	2.6	12.1	0.084 {694}
RF9 (inorganic sludge)	2.7[a]	<14.0	4.3	4.5	0.949[a] {6,683}
RF10 (organic sludge)	4.5[a]	<14.0	6.4	5.0	0.085[a] {607}
RF11 (organic sludge)	3.8[a] 4.9[b]	<14.0	4.1	3.0	0.148[a] {643} 0.114[b] {496}
SG5 (organic sludge)	4.1	<14.0	6.8	6.8	0.077 {508}
RF12 (MSE salts)		<7.0			15.02 {4.8E5}
RF13 (MSE salts)	2.7	<7.0	8.9	8.3	73.77 {9.9E5}

Table 15. (continued).

Test Sample	%RSD [(s/μ) x 100] per Test Sample TRU α (Ci) Configuration	Precision QAO % RSD on a (Ci)	%RSD [(s/$\bar{x}$) x 100] per Test Sample TRU α (Ci) Configuration	%RSD [(s/$\bar{x}$ x 100)] per Test Sample ^{239}Pu Mass	Test Sample Total α Activity (Ci) {nCi/g}
SG6 (MSE salts)	13.3	<7.0	19.5	2.6	5.921 {8.7E4}
SG7 (MSE salts)	2.2	<7.0	2.4	3.3	4.399 {7.8E4}
RF14 (glass)	6.4	<14.0	7.3	1.7	0.182 {2,141}
RF15 (glass)	1.3	<14.0	1.5	0.9	0.238 {4,091}
RF16 (glass)		<7.0			7.783 {2.0E5}
SG8 (glass)	1.5	<14.0	1.3	1.1	0.541 {5,578}
RF17 (raschig ring)	1.8	<14.0	2.1	1.2	0.087 {1,323}
RF18 (raschig ring)	1.8	<14.0	1.6	1.6	0.615 {1.3E4}
SG9 (raschig ring)	2.6	<14.0	2.8	1.6	0.0796 {1.2E3}
RF19 (mixed metals)		blank			0
RF20 (mixed metals)	2.5	<14.0	2.6	0.6	0.437 {3,624}
RF21 (mixed metals)	7.7	<14.0	7.8	7.2	1.575 {2.4E4}
SG10 (mixed metals)	0.7	<14.0	1.2	1.6	1.0464 {1.8E4}
SG11 (zero matrix)	5.7	<14.0	5.2	7.0	0.0422

a. SAS active mode basis.
b. Radiochemistry data basis.

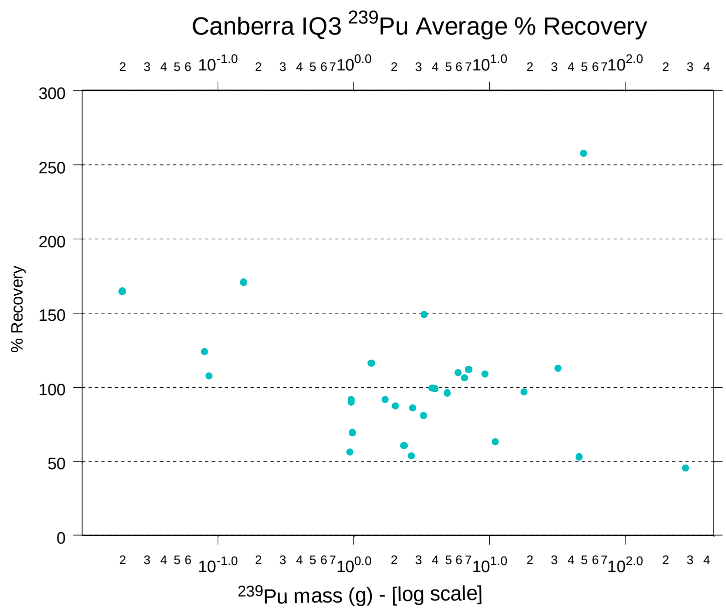

Figure 9. Plot of % Recovery as a function of ^{239}Pu mass for the Canberra Industries IQ3 System.

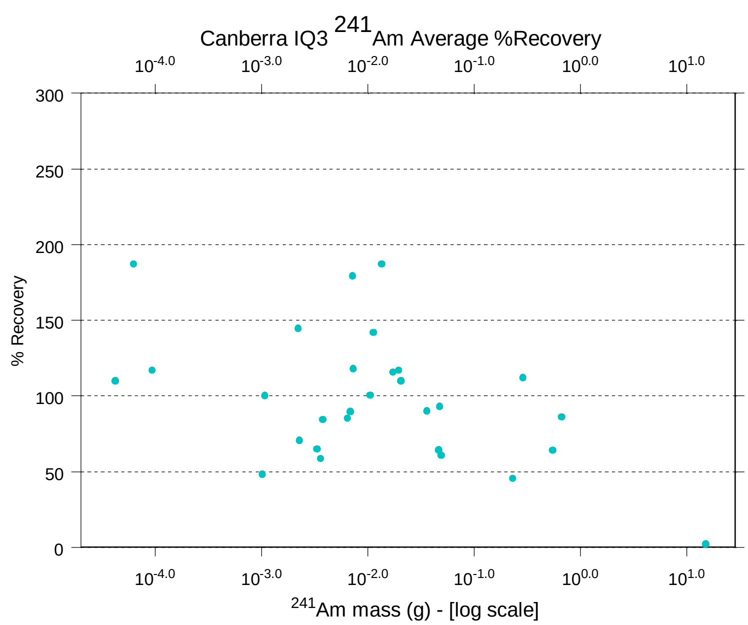

Figure 10. Plot of % Recovery as a function of ^{241}Am mass for the Canberra Industries IQ3 System.

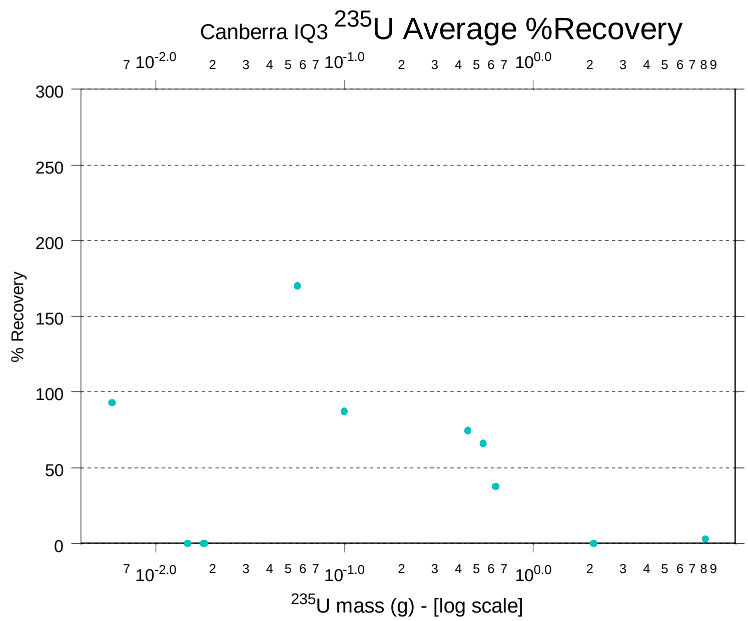

Figure 11. Plot of % Recovery as a function of ^{235}U mass for the Canberra Industries IQ3 System.

4.4.5 Los Alamos National Laboratory - Tomographic Gamma Scanner (TGS)

The bias results for TGS are presented in Tables 16 and 17. Corresponding relative precision and MDC data are reported in Table 18. Figures 12, 13, and 14 present average percent recovery as a function of radionuclide mass. The preliminary CEP test results tabulated below are based on measurements acquired by the LANL TGS mobile assay system using the WIN_TGS, Version 2.20, data acquisition and TGS_ARC, Version 1.1, data reduction software packages. The LANL TGS system as configured for the CEP test series also included the FRAM isotopics system. All CEP evaluations and results for the LANL TGS mobile assay system apply to this declared hardware/software configuration.

Table 16. LANL TGS bias results (surrogate test samples).

Surrogate Test Samples	Total α Avg %R ($\bar{\bar{x}}/\mu$)	Total α % Recovery Acceptance Criteria (95% Confidence Bounds)		^{239}Pu Avg (%R)	^{241}Am Avg (%R)	^{235}U Avg (%R)	^{238}U Avg (%R)
		Lower % R	Upper %R				
Graphite (SG1)	412.5	100.9	114.1	281.5	1,324.0	—	—
Combustibles (SG2)	104.0	32.1	197.9	103.0	118.1	—	—
Filters/insul (SG3)	93.0	50.9	149.1	94.6	93.2	—	—
Inorganic sludge (SG4)	1,907.0	425.6	-195.6	1,871.0	1,842.0	0.0	0.0
Organic sludge (SG5)	93.7	56.6	173.4	86.6	169.5	—	—
MSE salts (SG6)	109.2	51.8	148.2	92.9	138.0	—	—
MSR salts (SG7)	87.1	55.2	144.8	75.6	103.4	—	—
Glass (SG8)	88.8	32.9	197.1	86.6	95.7	—	—
Raschig rings (SG9)	91.6	36.7	193.3	91.2	98.9	—	—
Metals (SG10)	85.8	35.1	194.9	119.6	76.6	—	—
Empty (SG11)	238.3	104.9	95.1	280.5	209.8	—	—

Table 17. LANL TGS first pass bias results (actual waste test samples).

Actual Rocky Flats Test Samples	Total α Avg %R ($\bar{x}/\mu$)	Total α Recovery Acceptance Criteria (95% Confidence Bounds)		^{239}Pu Avg (%R)	^{241}Am Avg (%R)	^{235}U Avg (%R)	^{238}U Avg (%R)
		Lower % R	Upper %R				
Graphite (RF1)	111.3	33.9	196.1	109.1	146.7	—	—
Graphite (RF2)	115.0	33.6	196.4	113.2	125.8	—	—
Combustibles (RF3)	83.2	33.9	196.1	83.4	92.2	71.3	—
Combustibles (RF4)	1,617.0	620.0	-405.0	982.0	3,212.0	0.0	
Filters (RF5)	170.5	39.0	191.0	171.7	204.6	0.0	
Filters (RF6)	45.5	50.4	149.6	44.9	42.4	—	—
Inorganic sludge (RF7)[a]	276.8	104.8	95.2	317.3	274.1	0.0	0.0
Inorganic sludge (RF7)[b]	397.1	108.6	121.4	376.8	394.5	0.0	—
Inorganic sludge (RF8)[a]	103.0	47.5	182.5	103.6	102.1	101.1	0.0
Inorganic sludge (RF8)[b]	100.0	47.0	183.0	91.8	108.0	101.1	—
Inorganic sludge (RF9)[a]	69.0	34.7	195.3	68.4	66.3	—	—
Inorganic sludge (RF9)[b]							
Organic sludge (RF10)[a]	95.6	39.7	190.3	96.8	68.1	0.0	—
Organic sludge (RF10)[b]							
Organic sludge (RF11)[a]	120.5	46.0	184.0	116.5	113.9	0.0	—
Organic sludge (RF11)[b]	156.2	50.7	179.3	147.7	195.1	0.0	—
MSE (RF12)	100.4	52.5	147.5	100.1	110.5	—	—

Table 17. (continued).

Actual Rocky Flats Test Samples	Total α Avg %R ($\bar{\bar{x}}/\mu$)	Total α Recovery Acceptance Criteria (95% Confidence Bounds) Lower % R	Upper %R	^{239}Pu Avg (%R)	^{241}Am Avg (%R)	^{235}U Avg (%R)	^{238}U Avg (%R)
MSE (RF13)	20.4	50.5	149.5	30.9	26.7	0.0	—
Glass (RF14)	134.0	35.9	194.1	136.0	127.4	129.4	—
Glass (RF15)	90.7	33.8	196.2	92.6	94.3	119.1	—
Glass (RF16)							
Raschig ring (RF17)							
Raschig ring (RF18)							
Metals (RF19)[c]							
Metals (RF20)							
Metals (RF21)							

a. SAS active mode basis.
b. Radiochemistry data basis.
c. Blank, no detectable activity.

Table 18. LANL TGS relative precision/MDC bias data.

Test Sample	%RSD [(s/μ) x 100)] per Test Sample TRU α (Ci) Configuration	Precision QAO %RSD on α (Ci)	%RSD [(s/$\bar{x}$) x 100] per Test Sample TRU α (Ci) Configuration	% RSD [(s/$\bar{x}$ x 100)] per Test Sample ^{239}Pu (g) Measure	Test Sample Total α Activity (Ci) {nCi/g}
RF1 (graphite)	4.6	<14.0	4.1	4.1	0.5029 {1.4E4}
RF2 (graphite)	4.3	<14.0	3.8	3.7	0.7986 {1.2E4}
SG1 (graphite)	72.5	<18.0	17.6	17.7	0.0071 {73}
RF3 (dry combustibles)	4.7	<14.0	5.6	5.6	0.4313 {1.3E4}
RF4 (dry combustibles)	690.0	<18.0	42.7	42.7	0.0018 {56}
SG2 (dry combustibles)	2.5	<14.0	2.4	2.3	0.3144 {7,149}
RF5 (filters/insulation)	6.3	<14.0	6.3	6.3	0.2847 {4,825}
RF6 (filters/insulation)	0.4	<7.0	4.7	4.7	11.998 {4.0E5}
SG3 (filters/insulation)	1.1	<7.0	1.2	1.2	4.8906 {1.22E5}
RF7 (inorganic sludge)	65.3[a] 93.6[b]	<7.0 <14.0	23.6	23.4	2.564[a] {1.7E4} 1.79[b] {1.2E4}
RF8 (inorganic sludge)	20.8[a] 20.8[b]	<14.0	20.2	20.2	0.353[a] {1782} 0.364[b] {1,838}
SG4 (inorganic sludge)	471.0	<14.0	24.7	24.5	0.0836 {694}
RF9 (inorganic sludge)	5.6	<14.0	8.1	8.2	0.949[a] {6,683}
RF10 (organic sludge)	12.0	<14.0	12.0	12.0	0.085[a] {607}
RF11 (organic sludge)	19.0[a] 24.7[b]	<14.0	15.8	15.8	0.148[a] {643} 0.114[b] {496}
SG5 (organic sludge)	31.7	<14.0	33.8	33.7	0.0774 {508}

Table 18. (continued).

Test Sample	%RSD [(s/μ) x 100)] per Test Sample TRU α (Ci) Configuration	Precision QAO %RSD on α (Ci)	%RSD [(s/$\bar{x}$) x 100] per Test Sample TRU α (Ci) Configuration	% RSD [(s/$\bar{x}$ x 100)] per Test Sample ^{239}Pu (g) Measure	Test Sample Total α Activity (Ci) {nCi/g}
RF12 (MSE salts)	3.0	<7.0	2.9	3.0	15.019 {4.8e5}
RF13 (MSE salts)	0.6	<7.0	2.9	2.4	73.765 {9.9E5}
SG6 (MSE salts)	2.1	<7.0	1.9	1.9	5.921 {8.7e4}
SG7 (MSE salts)	6.2	<7.0	7.1	7.1	4.399 {7.8e4}
RF14 (glass)	7.0	<14.0	5.2	5.2	0.182 {2,141}
RF15 (glass)	4.5	<14.0	4.9	5.0	0.2381 {4,091}
RF16 (glass)					7.783 {2.0E5}
SG8 (glass)	3.5	<14.0	3.9	3.9	0.5412 {5,578}
RF17 (raschig ring)					0.0872 {1,323}
RF18 (raschig ring)					0.6153 {1.3e4}
SG9 (raschig ring)	7.3	<14.0	12.0	8.0	0.0796 {1.2e3}
RF19 (mixed metals)			19.4	19.4	0.0
RF20 (mixed metals)					0.4367 {3,624}
RF21 (mixed metals)					1.5746 {2.4e4}
SG10 (mixed metals)	6.1	<14.0	7.1	7.1	1.0464 {1.8e4}
SG11 (zero matrix)	65.3	<14.0	27.4	28.0	0.0422

a. SAS active mode basis.
b. Radiochemistry data basis.

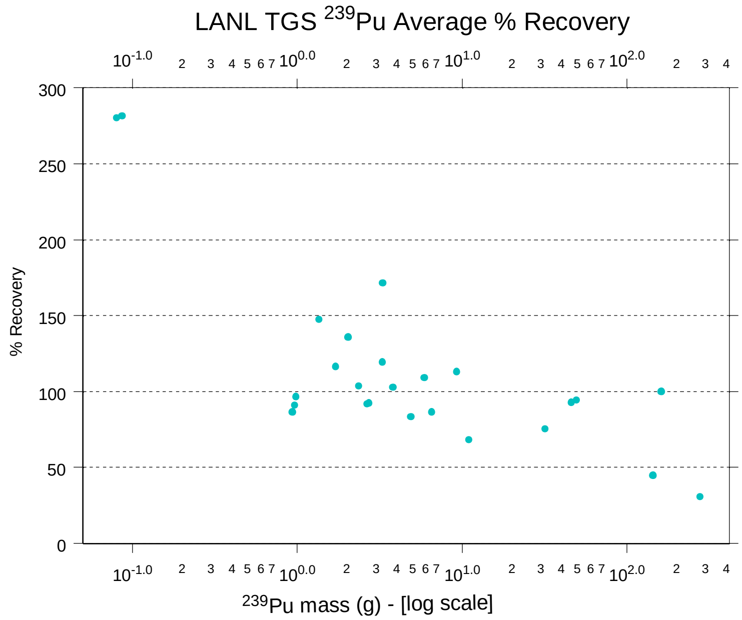

Figure 12. Plot of % Recovery as a function of ^{239}Pu mass for the Los Alamos National Laboratory TGS System.

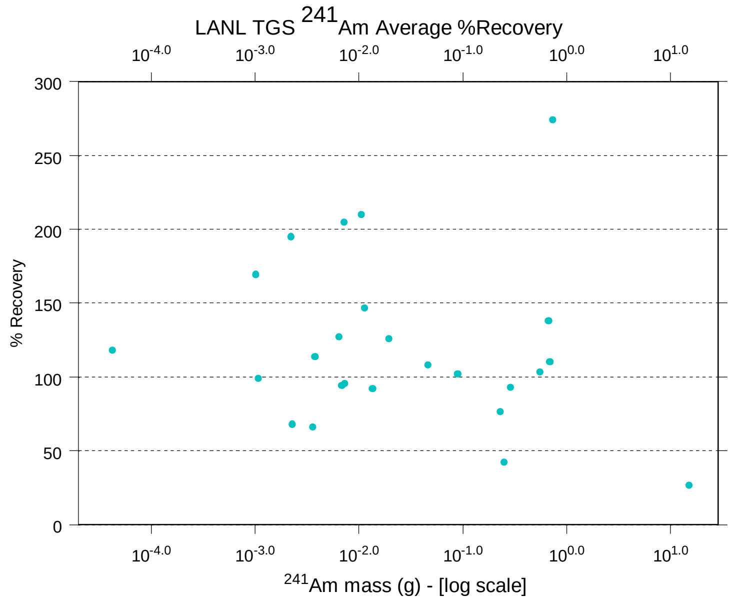

Figure 13. Plot of % Recovery as a function of ^{241}Am mass for the Los Alamos National Laboratory TGS System.

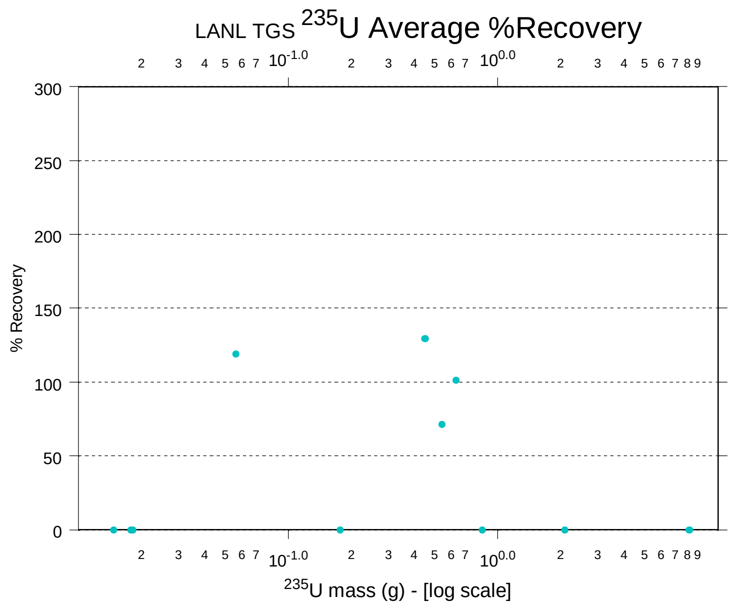

Figure 14. Plot of % Recovery as a function of ^{235}U mass for the Los Alamos National Laboratory TGS System.

5. DISCUSSION OF RESULTS

The CEP performance results can be interpreted and analyzed in a number of ways and will be addressed in detail in the CEP final report. Nevertheless, preliminary statements of performance can be made from simple examination of the bias and relative precision tables and the plots of average percent recovery versus radionuclide mass. For test samples comprised of nominal weapons grade plutonium at masses yielding statistically significant data, there are relatively few total bias and relative precision values outside the respective acceptance ranges for the primary radionuclide of quantification. These radionuclides are ^{239}Pu for gamma and ^{240}Pu for neutron based technologies. There is noted biases and high relative precision associated with low alpha activity concentration test samples, i.e., near 100 nCi/g. Data associated with test samples identified as having more than one interfering attribute displays a greater frequency of larger bias and relative precision values.

Several test samples were indicated in Section 3.3 as possessing attributes posing complications to existing waste NDA technologies. Discussion of these test samples are ordered per the number of complicating attributes per test sample.

Two test samples, SG1 and RF4, possess the one challenging attribute of low plutonium mass/activity (i.e., segregation at 100 nCi/g). The matrix attributes of either SG1 or RF4 do not present significant system complication nor does the radionuclidic composition, although RF4 does have a minute quantity of ^{235}U. All systems that processed the SG1 and RF4 test samples had unacceptable percent recovery on total TRU alpha activity. The only exception resulted where an allowance was granted to allow a longer count time on SG1. The TRU alpha activity concentration of SG1 was 73 nCi/g, higher than the 60 nCi/g QAPP MDC requirement. A similar situation was observed for the RF4 test sample at an estimated alpha activity concentration of 56 nCi/g. It must be noted that a longer measurement time may have provided for acceptable results. This is an important point in that routine counting times, on the order of 30 minutes, do not appear sufficient to accommodate LLW/TRU segregation at the necessary confidence level.

Three test samples, RF3, RF14, and SG10, possess the one challenging attribute of nonstandard weapons grade plutonium radionuclidic distributions. All participants that processed these samples had acceptable total alpha activity percent recovery and relative precision. Although there are no requirements for percent recovery on individual radionuclide mass basis, the ^{241}Am and ^{235}U mass recoveries were reasonable for all measurement systems. It is important to note that these test samples possess only one potentially confounding attribute and do not have any other major interference factors such as high density.

Results on the two samples, SG11 and SG4, demonstrate that multiple attributes within the same test sample can impact system performance. The SG4 inorganic sludge surrogate possesses a high density matrix at 1.1 g/cm^3, other radionuclides in addition to weapons grade plutonium radionuclides, and a relatively low plutonium mass loading of 154 milligrams. For most participants, significant error is associated with the determination of one or more of the ^{241}Am, ^{235}U, and ^{238}U mass values for the SG4 test sample. Two of the four participants had unacceptable alpha activity percent recovery and primary plutonium mass determination.

Unacceptable results associated with the SG4 test sample differ as a function of technology type. Two of the gamma based systems properly quantified the ^{239}Pu and ^{241}Am masses, but failed completely on the ^{235}U and ^{238}U mass determination. These two systems appear to have adequate capability for addressing matrix losses as well as sufficient sensitivity. There is, however, a problem with complete radionuclidic identification and quantification. The third gamma based system was unable to quantitate the ^{239}Pu mass as well as the ^{241}Am, ^{235}U, and ^{238}U masses. This appears to be related to a lack of sensitivity with possible inability to accommodate the dense matrix. The neutron based system was biased low on ^{240}Pu and did not yield adequate recoveries on the ^{241}Am, ^{235}U, and ^{238}U masses. This indicates insufficient accounting of matrix losses and poor radionuclidic distribution measurement. All but one of the systems produced acceptable total alpha activity relative precision. Without a detailed analysis of the measurement data acquired for each system, it is difficult to state the precise cause of the poor performance. This detailed analysis will be reported on in the CEP final report. It is clear, however, that the presence of multiple complicating attributes, e.g., high density coupled with the presence of additional TRU radionuclides results in less than satisfactory performance.

The zero matrix drum SG11 contained a relatively low loadings of plutonium, 80 milligrams of ^{239}Pu, and an elevated ^{241}Am to plutonium mass ratio. One of the four participants produced acceptable total alpha activity percent recovery and relative precision. Two of the four systems displayed reasonable capability to identify and quantify the primary plutonium isotope and reasonable ^{241}Am recoveries. Overall, less than satisfactory performance is observed when more than one complicating attribute is present, i.e., low plutonium mass and a radionuclidic distribution deviating from nominal weapons grade plutonium. It should be noted that three of the four systems have stated sensitivities for weapons grade plutonium below the 80 milligram loading of the SG11 test sample indicating some degree of ^{241}Am interference and lack of proper radionuiclidic distribution determination.

6. CONCLUSIONS

The CEP was conducted in accordance with the test plan, yielding a wealth of objective data on technology-specific system performance. The program afforded the participants access to NIST traceable radioactive standards, surrogate matrix drums, and actual TRU waste drums. This enabled several participants to make significant enhancements to their respective systems and supported all participants in attaining CAO certification authority.

The goal of the CEP was to objectively establish a known and unbiased waste NDA data and information base. Based on the data and information collected, it can be stated, with appropriate qualifications, that a general state of acceptable capability exists. However, there is also evidence of technology deficiencies, particularly when the number of complication attributes is two or more. The effect of combinations of attributes will be documented in detail in the CEP final report.

The CEP data indicate a general state of waste NDA proficiency for waste configurations exhibiting reasonable matrix densities and radionuclide mass loadings. Techniques employed account for matrix effects appear sufficiently developed for waste forms exhibiting reasonable densities, e.g., less than 0.6 g/cm^3. Evaluated technologies are sufficient to characterize waste with radioactive material mass loadings comprised of nominal weapons grade plutonium sufficient to yield statistically significant data. This indicates that in general, calibration, data acquisition, and reduction techniques under such conditions are adequate with respect to the bias and relative prevision QAOs.

There is an apparent issue regarding detection sensitivity and compliance with the MDC QAO criteria. Although detection limits are a function of background counts, other interference sources, matrix or radionuclide based, contribute to the actual achievable MDC. Performance, with respect to the CEP MDC estimatation, total bias, and relative precision parameters, for many test samples indicates that the overall system MDC is well above the QAPP 60 nCi/g criteria. To a certain extent this can be mitigated through the use of longer measurement times. In other instances, there does not appear to be adequate accounting of interferences affecting MDC such that increased measurement times leads to diminishing returns. Also, the use of increased measurement time is for the most part undesirable from a production standpoint.

Finally, the ability to yield acceptable bias and precision performance is, under many circumstances, compromised by the number of complicating attributes inherent in the waste matrix configuration. For instance, when the radionuclic distribution departs from that associated with nominal weapons grade plutonium, there is reasonable capability to correctly determine the mass of the various nuclides. If this configuration is compounded by a low mass, the primary plutonium deteriorates. If this configuration is again compounded with a high density matrix, the ability to perform in an acceptable manner in further reduced.

In summary, the CEP achieved the stated end-user support objective. The data indicate that the NDA systems evaluated have a definite capability to perform assay of contact-handled TRU waste packaged in 55-gallon drums. There is, however, a performance envelop where this capability exists, an area near the boundaries where it is questionable, and a realm outside the envelope where the technologies do not perform. Therefore, the end user must be aware of this envelope and ensure the appropriate technology is selected. This program provides the end user with the waste type-specific performance data to assist in the assessment and selection of a given waste NDA technology.

7. REFERENCES

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